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(54) Title: BIODEGRADABLE ORIENTED POLYESTER FILM AND PREPARATION METHOD THEREOF

(57) Abstract: A biodegradable oriented polyester film comprising a random copolymer of i) a polycaprolactone having a number average molecular weight of 500 to 10,000 and/or a caprolactone monomer and ii) a lactide is environmentally friendly and exhibits excellent transparency and maximized flexibility without bleed out problem, and therefore can be used for various packaging and wrapping uses.



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DESCRIPTION
BIODEGRADABLE ORIENTED POLYESTER FILM
AND PREPARATION METHOD THEREOF

5 **FIELD OF THE INVENTION**

The present invention relates to a biodegradable oriented polyester film, which is useful for packaging, and a method for the preparation thereof.

10 **BACKGROUND OF THE INVENTION**

Conventional plastics such as polyvinyl chloride (PVC), polyethylene (PE) and polypropylene (PP) are not completely satisfactory in terms of their performance characteristics. For example, they generate toxic pollutants when
15 incinerated, and emit large amounts of carbon dioxide contributing greenhouse effect. Further, such plastic films are not biodegradable and accumulate in the soil when disposed.

Recently, a number of countries have implemented or consider implementing carbon taxes or emission trading schemes for reducing greenhouse
20 gas emissions.

In order to solve such problems, there have been conducted a number of studies on biopolymer derived from biomass and biodegradable aliphatic polyesters, particularly polylactic acid films. Although such films have satisfactory mechanical properties and transparency, they have poor flexibility due
25 to high crystallinity, which is them undesirable for packaging. In case of cold storage or frozen food storage, the packaging films easily break during storage and handling due to insufficient pin-hole resistance.

Japanese Laid-open Patent Publication No. 2003-147098 discloses a biodegradable polylactic acid film excellent transparency and flexibility, and
30 moldable with a T-die extruder. According to this patent publication, when the

content of D-lactic acid in polylactic acid is more than 4 parts by weight, adhesion of the film to a casting roll occurs. Thus, a ternary system polylactic acid, poly(butylene adipate/terephthalate)(PBAT) and polycaprolactone has been used to form films. However, haze of the obtained film is too high due to poor
5 miscibility of resins thereby opaque films are obtained despite excellent transparency.

Japanese Laid-open Patent Publication No. 2004-244553 discloses a flexible and biodegradable polyester laminated film comprising a terpolymer of butanediol/succinic acid/caprolactone and amorphous polylactic acid, as a low
10 temperature heat-sealing film. Further, in order to improve tear strength and heat sealing strength of the film disclosed in the above patent publication, Japanese Laid-open Patent Publication No. 2004-237473 discloses a biodegradable polyester laminated film which comprises an aromatic polyester copolymer of PBAT/1,4-butanediol and a copolymer of aliphatic polyester copolymer of
15 succinic acid/1,4-butanediol/caprolactone. However, the film does not have enough flexibility.

Thus, there is still a need for a novel biodegradable film having enough transparency and flexibility.

20 SUMMARY OF THE INVENTION

Accordingly, in accordance with one aspect of the present invention, there are provided a biodegradable oriented polyester film having enough flexibility and high transparency and a preparation method thereof.

25 In accordance with another aspect of the present invention, there is provided a biodegradable oriented polyester film comprising a random copolymer of i) a polycaprolactone having a number average molecular weight of 500 to 10,000 and/or a caprolactone monomer and ii) a lactide.

In accordance with a further aspect of the present invention, there is
30 provided a process for preparing a biodegradable oriented polyester film

comprising the steps of: (a) random copolymerizing i) a polycaprolactone having a number average molecular weight of 500 to 10,000 and/or a caprolactone monomer and ii) a lactide to obtain a copolymer resin; (b) melt-extruding the copolymer resin to obtain a non-oriented sheet; and (c) orienting the non-oriented sheet in at least one direction, and heat-setting the resulting sheet to obtain the biodegradable oriented polyester film.

DETAILED DESCRIPTION OF THE INVENTION

10 In accordance with one embodiment of the present invention, the biodegradable oriented polyester film is formed by random copolymerization of i) a polycaprolactone having a number average molecular weight of 500 to 10,000 and/or a caprolactone monomer and ii) a lactide.

A polycaprolactone is prepared by a ring-opening reaction of caprolactone monomers having a long alkyl chain (5 CH₂), thereby giving enough flexibility to the inventive film. Accordingly, flexibility of the film can be maximized by using a caprolactone monomer and/or a polycaprolactone having a number average molecular weight of 500 to 10,000 as an ingredient of a copolymer.

In the film of the present invention, ingredient i) is preferably a polycaprolactone having a number average molecular weight of 500 to 10,000.

The lactide of ingredient ii) is preferably L-lactide. The film shows more improved physical properties when L-lactide of high purity is used.

The weight ratio of ingredients i) and ii) comprised in the film is preferably 80:20 to 5:95.

25 The heat shrinkage of the film in either the longitudinal or transverse direction after heat treatment in hot air at 100 °C for 5 min is preferably 1 to 75%.

The film preferably exhibits an initial elastic modulus of 50 to 350 kgf/mm².

The film preferably exhibits a haze of 20% or less.

30 The film preferably has a thickness of 10 to 300 μm.

The biodegradable oriented polyester film of the present invention is prepared by a process comprising the steps of: (a) random copolymerizing i) a polycaprolactone having a number average molecular weight of 500 to 10,000 and/or a caprolactone monomer and ii) a lactide to obtain a copolymer resin; (b) melt-extruding the copolymer resin to obtain a non-oriented sheet; and (c) orienting the non-oriented sheet in at least one direction of the longitudinal and transverse directions, and heat-setting the resulting sheet to obtain the biodegradable oriented polyester film.

In step (a), ingredients i) and ii) are reacted in a weight ratio ranging from 80:20 to 5:95, and the random copolymerization is preferably carried out at a temperature of 170~190 °C for 3~5 hours under inert atmosphere, e.g., under nitrogen atmosphere.

For example, the random copolymer resin may be prepared by a process comprising the steps of mixing 5~80 parts by weight of ingredient i) and 20~95 parts by weight of ingredient ii), adding thereto 0.01~0.1 part by weight of tin catalyst(stannous octoate) and 0.1~0.2 part by weight of SiO₂ as an antiblocking agent, and reacting the mixture at a temperature of 170~190 °C for 3~5 hours under nitrogen atmosphere, and then removing by-products or unreacted materials under reduced pressure.

A melt-extruding temperature in step (b) is preferably 180~280 °C.

Further, in step (c), the longitudinal drawing and/or the transverse drawing are preferably performed at a draw ratio of 2.5 to 3.5 and 3.5 to 4.5, respectively, and the heat-setting temperature is preferably 80~170 °C.

In accordance with the present invention, there is also provided a packaging material comprising the biodegradable oriented polyester film of the present invention.

The inventive film is environmentally friendly with high biodegradability

and exhibits excellent transparency (a light transmittance of at least 90% and a haze of 20% or less). Further, the inventive film is made of polylactic acid-caprolactone copolymer resin thereby secures enough flexibility. That is, flexibility of the film can be maximized by using a caprolactone monomer and/or a polycaprolactone having a number average molecular weight of 500 to 10,000 as ingredients of copolymerization, without bleed out problems related with liquid plasticizers. Further, the inventive film is produced via drawing (orientation), so it has excellent strength and elongation characteristics.

The inventive film can be used for various packaging and wrapping uses, e.g., as a general packaging film, a fancy packaging film, a general label, a heat-shrinkable label, an agricultural mulching film and an overwrapping film, and for laminating on a paper, laminating on non-woven fabric, preparing a disposable gown, laminating on wallpaper and laminating on flooring.

EXAMPLES

The following examples are now given for the purpose of illustration only, and are not intended to limit the scope of the invention.

The following materials are used in Examples:

- Caprolactone monomer: CapaTM Monomer, Perstorp Inc.
- L-lactide: Musashino Chemical Laboratory Ltd.
- Tin catalyst: Stannous octoate, TEGOKAT, Goldschmidt GmbH.
- Polycaprolactone(Mn 1,000~10,000): PL1000~PL10,000, Perstorp Inc.
- Polylactic acid resin: 4032D, NatureWorks LLC.

Example 1

15 parts by weight of caprolactone monomer, 85 parts by weight of L-lactide, 0.02 part by weight of tin catalyst, and 0.2 part by weight of SiO₂ as an

antiblocking agent were mixed together. The mixture was reacted at a temperature of 190°C for 5 hours under nitrogen atmosphere, and then by-products or unreacted materials were removed under reduced pressure, to obtain a random copolymer resin (Mn 100,000).

5 The resulting copolymer resin was melt-extruded at 200°C and the resulting sheet was drawn on a casting roll kept at 10°C at a draw ratio of 3.0 in the longitudinal direction at 85°C and at a draw ratio of 4.0 in the transverse direction at 105°C, and then, heat set at 100°C to obtain an oriented polyester film having a thickness of 20 µm.

10 **Example 2**

 The procedure of Example 1 was repeated except for heat setting temperature of 160°C to obtain an oriented polyester film having a thickness of 20 µm.

Example 3

25 25 parts by weight of polycaprolactone (Mn 1,000), 75 parts by weight of L-lactide, 0.02 part by weight of tin catalyst, and 0.2 part by weight of SiO₂ as an antiblocking agent were mixed together. The mixture was reacted at a temperature of 180°C for 5 hours under nitrogen atmosphere, and then by-products or unreacted materials were removed under reduced pressure to obtain a random copolymer resin (Mn 90,000).

30 The resulting copolymer resin was melt-extruded at 210°C and the resulting sheet was drawn on a casting roll kept at 9°C at a draw ratio of 3.0 in the longitudinal direction at 80°C and at a draw ratio of 4.0 in the transverse direction at 100°C, and then, heat set at 105°C to obtain an oriented polyester film having a thickness of 20 µm.

Example 4

The procedure of Example 3 was repeated except for heat setting temperature of 150°C to obtain an oriented polyester film having a thickness of 20 µm.

Example 5

20 parts by weight of polycaprolactone (Mn 3,000), 80 parts by weight of L-lactide, 0.025 part by weight of tin catalyst, and 0.2 part by weight of SiO₂ as an antiblocking agent were mixed together. The mixture was reacted at a temperature of 180°C for 5 hours under nitrogen atmosphere, and then by-products or unreacted materials were removed under reduced pressure to obtain a random copolymer resin (Mn 110,000).

The resulting copolymer resin was melt-extruded at 200°C and the resulting sheet was drawn on a casting roll kept at 15°C at a draw ratio of 3.0 in the longitudinal direction at 77°C and at a draw ratio of 3.8 in the transverse direction at 100°C, and then, heat set at 95°C to obtain an oriented polyester film having a thickness of 20 µm.

Example 6

23 parts by weight of polycaprolactone (Mn 7,000), 77 parts by weight of L-lactide, 0.03 part by weight of tin catalyst, and 0.2 part by weight of SiO₂ as an antiblocking agent were mixed together. The mixture was reacted at a temperature of 200°C for 6 hours under nitrogen atmosphere, and then by-products or unreacted materials were removed under reduced pressure to obtain a random copolymer resin (Mn 95,000).

The resulting copolymer resin was melt-extruded at 180°C and the resulting sheet was drawn on a casting roll kept at 13°C at a draw ratio of 3.0 in

the longitudinal direction at 77°C and at a draw ratio of 3.8 in the transverse direction at 100°C, and then, heat set at 95°C to obtain an oriented polyester film having a thickness of 20 µm.

5 Example 7

7 parts by weight of caprolactone, 10 parts by weight of polycaprolactone (Mn 1,000), 83 parts by weight of L-lactide, 0.03 part by weight of tin catalyst, and 0.2 part by weight of SiO₂ as an antiblocking agent were mixed together.
10 The mixture was reacted at a temperature of 190°C for 5 hours under nitrogen atmosphere, and then by-products or unreacted materials were removed under reduced pressure to obtain a random copolymer resin (Mn 80,000).

The resulting copolymer resin was melt-extruded at 190°C and the resulting sheet was drawn on a casting roll kept at 12°C at a draw ratio of 3.0 in
15 the longitudinal direction at 85°C and at a draw ratio of 4.0 in the transverse direction at 100°C, and then, heat set at 85°C to obtain an oriented polyester film having a thickness of 20 µm.

Comparative Example 1

20

15 parts by weight of polyethylene glycol, 85 parts by weight of L-lactide, 0.004 part by weight of tin catalyst, and 0.1 part by weight of SiO₂ as an antiblocking agent were mixed together. The mixture was reacted at a temperature of 170°C for 6 hours under nitrogen atmosphere, and then by-
25 products or unreacted materials were removed under reduced pressure to obtain a random copolymer resin of Mn 80,000.

The resulting copolymer resin was melt-extruded at 190°C and the resulting sheet was drawn on a casting roll kept at 15°C at a draw ratio of 3.0 in the longitudinal direction at 90°C and at a draw ratio of 4.0 in the transverse
30 direction at 110°C, and then, heat set at 160°C to obtain an oriented polyester film

having a thickness of 20 μm .

Comparative Example 2

5 14 parts by weight of polyol having a trimethylene group in the repeating units, 86 parts by weight of L-lactide, 0.01 part by weight of tin catalyst, and 0.2 part by weight of SiO_2 as an antiblocking agent were mixed. The mixture was reacted at a temperature of 190°C for 7 hours under nitrogen atmosphere, and then by-products or unreacted materials were removed under reduced pressure to
10 obtain a random copolymer resin of Mn 70,000.

 The resulting copolymer resin was melt-extruded at 200°C and the resulting sheet was drawn on a casting roll kept at 12°C at a draw ratio of 3.0 in the longitudinal direction at 85°C and at a draw ratio of 4.0 in the transverse direction at 105°C, and then, heat set at 150°C to obtain an oriented polyester film
15 having a thickness of 20 μm .

Comparative Example 3

 A polylactic acid resin(T_m 170°C, T_g 65°C) was dried with a hot air drier
20 at 100°C for 3 hours to remove moisture therefrom. 90 parts by weight of the dried resin, 10 parts by weight of caprolactone, and 0.1 part by weight of SiO_2 as an antiblocking agent were uniformly compounded using a twin screw extruder at 200°C, to obtain a homogeneous compound.

 The resulting compound was melt-extruded at 190°C and the resulting
25 sheet was drawn on a casting roll kept at 13°C at a draw ratio of 3.0 in the longitudinal direction at 75°C and at a draw ratio of 4.0 in the transverse direction at 95°C, and then, heat set at 150°C to obtain an oriented polyester film having a thickness of 20 μm .

30 Compositions and preparation processes of the films of the Examples and

the Comparative Examples are shown in Table 1.

Performance Test

5 The films prepared in the Examples and the Comparative Examples were examined to determine the following properties thereof. The results are shown in Table 1.

(1) Molecular Weight

10

0.003 g of a copolymer resin was dissolved in tetrahydrofuran(THF) and the resulting solution was injected into a GPC with ELSD (Waters Ltd., USA) at room temperature, and the eluent rate was 1mL/min.

15 (2) Film Composition

A film sample was dissolved in a mixture of deuteriochloroform (CDCl_3) and trifluoroacetic acid (4:1 by weight ratio) and then analyzed with H-NMR (JSM-LA300, JEOL Ltd., Japan).

20

(3) Bleed Out

A film sample having a size of 20 cm x 20 cm was kept in a hot air oven at 150 °C for 2 hours, and evaluated as below.

25

○ : The film surface was not slippery, and causes blocking with each other.

× : The film surface was slippery, and does not cause blocking with each other.

30 (4) Initial Elastic Modulus, Strength and Elongation

The initial elastic modulus (kgf/mm²), the strength and the elongation were measured according to ASTM D 882 using a film sample of 100 mm x 15 mm with an elongation rate of 200 mm/min and an interval between chucks of 50 mm by using a universal tester (UTM 4206-001, Instron Inc.). The lower initial elastic modulus indicates the higher flexibility.

(5) Haze

A hazemeter (SEP-H, Nihon Semitsu Kogaku Co., Ltd., Japan) with a C-light source was used.

(6) Pin-hole Resistance

A sample film was subjected to a flex crack resistance test using Gelbo-Flex tester (Gelbo Inc., USA) consisting of a 165 mm sample fixing plate, a 88 mm sample fixing diameter and a 125 mm traveling distance, at a twisting motion of 450 degrees for 450 cycles for about 10 minutes.

Next, the tested sample film was placed on a white paper, whereon an oily nitroglycerin-based ink was applied with a doctor blade. The number of ink dots appeared on the white paper was counted as the pin-hole number. The results reported are the average of three repeats.

(7) Heat shrinkage (%)

A film sample was cut into a 200 mm (length) × 15 mm (width) piece, maintained at 100 °C in a circulating air oven for 5 minutes, and the change in the film length was measured. Using the following equation, the degrees of shrinkage in each of the longitudinal and transverse directions were calculated:

Heat shrinkage (%) = [(length before heat treatment – length after heat

treatment) / length before heat treatment] × 100

Table 1

	Item			Unit	Example							Comparative Example			
					1	2	3	4	5	6	7	1	2	3	
Component	Random copolymer	Caprolactone monomer		wt%	15	15						7			
		Polycaprolactone	Mn 1,000	wt%			25	25				10			
			Mn 3,000	wt%					20						
			Mn 7,000	wt%						23					
		Polyethylene glycol		wt%									15		
		Polyol		wt%										14	
	Compounding	Caprolactone monomer		wt%											10
Process	Extrusion Temp.			°C	200	200	210	210	200	180	190	190	200	190	
	Drawing Temp.	L.D. ¹⁾		°C	85	85	80	80	77	75	85	90	85	75	
		T.D. ²⁾		°C	105	105	100	100	100	96	100	110	105	95	
	Drawing ratio	L.D.		times	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	
		T.D.		times	4.0	4.0	4.0	4.0	3.8	3.7	4.0	4.0	4.0	4.0	
	Heat setting Temp.			°C	100	160	105	150	95	80	85	160	150	150	
Properties	Bleed out				X	X	X	X	X	X	X	X	X	○	
	Heat-shrinkage (100°C, 5 min)	L.D.		%	25	3	23	5	23	25	22	7	4	5	
		T.D.		%	30	5	29	7	35	30	27	10	6	7	
	Initial elastic modulus			kg f/mm ²	330	340	300	320	290	260	315	480	460	430	
	Strength			kg f/mm ²	17	18	19	22	18	23	18	17	20	21	
	Elongation			%	110	100	115	110	120	125	98	120	105	105	
	Pin-hole number			unit	50	55	45	48	43	38	48	100	90	85	
	Haze			%	3.80	4.20	7.60	8.20	6.50	7.50	5.80	5.50	6.00	4.50	
¹⁾ L.D.: longitudinal direction ²⁾ T.D.: transverse direction															

As shown in Table 1, the inventive films of Examples 1 to 7 showed superior properties over those of the films of Comparative Examples 1 to 3 in terms of bleed out, haze, pin-hole resistance, initial elastic modulus, and heat-shrinkability.

5 Especially, the polyester oriented films of Examples 3 to 7 employing a polycaprolactone having a number average molecular weight of 500~10,000 as the copolymer ingredient has maximized the film flexibility. More free volume between polylactic acid chains allows segments can move more easily.

10 The films of Examples 1, 3 and 5 to 7 had a high heat-shrinkability due to a low heat setting temperature. Accordingly, the films of Examples 1, 3 and 5 to 7 can be used as an overwrapping film, while the films of Examples 2 and 4 having a low heat-shrinkability are useful as a general packaging film.

15 In contrast, the films of Comparative Examples 1 and 2 made of polyethylene glycol (PEG) having a dimethylene group and polyol having a trimethylene group, have lower flexibility as compared with the inventive films of Examples using caprolactone having a pentamethylene group (5 CH₂).

20 Further, the film of Comparative Example 3 was prepared by extruding a compound/blend of caprolactone monomer and polylactic acid. The film exhibited bleed out problem, resulted from migration of caprolactone monomer having small molecular weight out to the film surface.

The films according to Examples and Comparative examples showed excellent strength and elongation because all of the films were subjected to a drawing process.

25 While the invention has been described with respect to the above specific embodiments, it should be recognized that various modifications and changes may be made to the invention by those skilled in the art which also fall within the scope of the invention as defined by the appended claims.

WHAT IS CLAIMED IS:

1. A biodegradable oriented polyester film comprising a random copolymer of i) a polycaprolactone having a number average molecular weight of 500 to 10,000 and/or a caprolactone monomer and ii) a lactide.
2. The biodegradable oriented polyester film of claim 1, wherein ingredient i) is a polycaprolactone having a number average molecular weight of 500 to 10,000.
3. The biodegradable oriented polyester film of claim 1, wherein the lactide is L-lactide.
4. The biodegradable oriented polyester film of claim 1, wherein ingredients i) and ii) are comprised in the random copolymer in a weight ratio ranging from 80:20 to 5:95.
5. The biodegradable oriented polyester film of claim 1, wherein the heat shrinkage of the film in either the longitudinal or transverse direction after heat treatment in hot air at 100 °C for 5 min is 1 to 75%.
6. The biodegradable oriented polyester film of claim 1, wherein the film exhibits an initial elastic modulus of 50 to 350 kgf/mm².
7. The biodegradable oriented polyester film of claim 1, wherein the film exhibits a haze of 20% or less.
8. The biodegradable oriented polyester film of claim 1, wherein the film has a thickness of 10 to 300 μ m.

9. A process for preparing a biodegradable oriented polyester film comprising the steps of:
- (a) random copolymerizing i) a polycaprolactone having a number average molecular weight of 500 to 10,000 and/or a caprolactone monomer and ii) a lactide to obtain a copolymer resin;
 - (b) melt-extruding the copolymer resin to obtain a non-oriented sheet; and
 - (c) orienting the non-oriented sheet in at least one direction of the longitudinal and transverse directions, and heat-setting the resulting sheet to obtain the biodegradable oriented polyester film.
10. The process of claim 9, wherein in step (a), ingredients i) and ii) are reacted in a weight ratio ranging from 80:20 to 5:95, and the random copolymerization is carried out at a temperature of 170~190 °C for 3~5 hours under inert atmosphere.
11. The process of claim 9, wherein the melt-extruding in step (b) is carried out at a temperature of 180~190 °C.
12. The process of claim 9, wherein in step (c), the longitudinal drawing and the transverse drawing are performed at a draw ratio of 2.5 to 3.5 and 3.5 to 4.5, respectively.
13. The process of claim 9, wherein the heat-setting in step (c) is carried out at a temperature of 80~170 °C.
14. A packaging material comprising the biodegradable oriented polyester film according to claim 1.