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(54) **Methods of packaging high-temperature materials and high-temperature packages thereof.**

(57) High-temperature materials which are liquid at elevated packing temperatures of 80° C or greater, are introduced in their liquid form into containers formed of polybutylene terephthalate (in film or molded form) at the elevated packaging temperature.

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METHODS OF PACKAGING HIGH-TEMPERATURE MATERIALS AND HIGH-TEMPERATURE PACKAGES THEREOF

The present invention relates to high-temperature packaging methods and to high-temperature packaging materials which are made of polybutylene terephthalate resins that are capable of being filled with fluids (i.e. liquids) at elevated temperatures without cooling the same.

Plastics have been widely used as packaging materials for liquid and solid foodstuffs which are supplied to the market in containers. These foodstuffs are usually subjected to high-temperature sterilization, such as in the case of natural fruit juice, cow's milk and the like, or are prepared at high-temperatures, such as in the case of chocolate products. However, when plastic materials are employed in high-temperature packaging applications, stronger physical characteristics are required than for plastics materials used in low-temperature packaging applications. More specifically, plastic materials employed in high-temperature packaging applications must usually exhibit high container strength characteristics at the packing temperatures, minimal (if any) container deformation due to heat shrinkage, no container "whitening" due to crystallization when heated, and high barrier properties (i.e., minimal transfer of the taste and/or odour from or to the container).

Conventional containers made of polyolefins, such as polyethylene or polypropylenes, ionomers, ethylene-vinyl acetate copolymers or the like, usually exhibit heat resistance but often deform when packaging occurs at high temperatures, particularly above 100° C. These high packaging temperatures also increase the transfer of a "polymer" odour to the packaged contents. As a result, polyolefin containers cannot usually be employed throughout the entirety of the high-temperature packaging process. For example, after high-temperature sterilization at temperatures between 130 to 135° C for several seconds in a sterilization container, cow's milk must first be cooled to about 60° C before being packaged in polyolefin containers. Similarly, chocolate must be packaged temporarily into disposable containers at a requisite packing temperature of between about 160 to 180° C, and then cooled before re-packaging within other containers. Such procedures are not only time-consuming, but also make the manufacturing process uneconomically more complicated.

The present invention is principally based upon the discovery that polybutylene terephthalate (PBT) resin exhibits excellent heat sealability in addition to excellent heat and oil resistance properties. Moreover, polybutylene terephthalate resin is substantially free from "polymer" taste and odour even at high temperatures typically encountered during packaging of foodstuffs. As a result, it has been found that polybutylene terephthalate is remarkably well suited for use as a packaging material for high-temperature packaging applications such that even a single layer film thereof has sufficient strength properties.

The present invention is therefore broadly concerned with the use of polybutylene terephthalate as a packaging material for high-temperature packaging applications, whereby containers of polybutylene terephthalate resin are filled with a high-temperature fluid (i.e., having a temperature of at least 80° C) during the packaging operation. The package of the invention comprises a container formed of polybutylene terephthalate, the contents of which had been placed within the container in a fluid form at temperatures of at least 80° C.

Specifically the present invention provides a method of packaging high-temperature materials comprising providing a material which is liquid at a packaging temperature of 80° C or greater, and then introducing the liquid material at the said packaging temperature into a container comprised of a polybutylene terephthalate resin. The present invention further provides such a method wherein the said liquid material is liquid at a packaging temperature of 100° C or greater and is introduced into the said container at the said packaging temperature of 100° C or greater.

The contents of the containers, such as aqueous and oily foodstuffs including drinks and beverages and non-foodstuffs, such as molten resins, waxes and paints can be introduced into a packing material of polybutylene terephthalate in fluid form at temperatures of 80° C or higher. The polybutylene terephthalate packaging material according to the present invention may be in the form of a bag, pouch or molded container, and may itself constitute the entire container, or may be used as an inner liner of non-polybutylene terephthalate containers.

These and other aspects of the invention will become more clear after careful consideration is given to the following detailed description of the preferred exemplary embodiments.

The polybutylene terephthalate resin that may be used in accordance with the present invention is a polyester prepared by the polycondensation of 1,4-butanediol with terephthalic acid or its lower alcohol ester, and may be in the form of a copolymer comprised of a major amount of polybutylene terephthalate.

The polybutylene terephthalate resin according to the present invention may optionally contain known

materials typically added to conventional thermoplastic and thermosetting resins to achieve desired performance characteristics provided that the characteristics required of the present invention are not deleteriously affected. If desired, a minor amount of another thermoplastic resin and/or inorganic filler may also be added to the polybutylene terephthalate base resin.

5 The term "packaging material" as used herein is intended to refer generically to bags, containers and the like. Bags can be produced by first forming a film from the polybutylene terephthalate resin by the inflation method or by the T-die method, and then fabricating the bags from the film by means of heat sealing or the like. On the other hand, containers can be produced by injection molding or other heat forming techniques, such as vacuum-forming films or sheets of polybutylene terephthalate resin.

10 Films of polybutylene terephthalate resin according to the present invention are most preferably formed using inflation or T-die film-forming methods. Particularly, films formed by the inflation method are preferred since they can be produced inexpensively and exhibit many physical advantages, including higher tensile strength and excellent gas barrier properties compared with films formed by the T-die method.

The inflation molded which is quite often employed to form polyethylene films, is a procedure whereby 15 a tubular film is formed. The T-die method is a procedure of extruding a plastics materials into a film through an extrusion molding die with a linear slit. Although the inflation method is overwhelmingly superior in terms of productivity, it is restricted in the types of resin materials that can be processed. Thus, the inflation method has not been universally appropriate for all types of resins. In fact, until recently, terephthalate resins have been thought to be unsuitable for use in inflation film forming. It has, however, 20 recently been discovered that specific polybutylene terephthalate resins, specifically those having a relatively high intrinsic viscosity of at least 1.0, preferably 1.0 to 2.5, are capable of being formed into films according to inflation film-forming techniques. See in this regard, our U.S Patent No. 4,869,864 issued on 26 September 1989. Such films exhibit physical properties favorably adapted for the purposes of the present invention as compared with conventional plastic films produced commercially.

25 In contrast, a polybutylene terephthalate resin having an intrinsic viscosity of less than 1.0 is difficult to form into a film no matter how the conditions of inflation may be varied. Where polybutylene terephthalate resins having an intrinsic viscosity of less than 1.0 must be used due to their physical properties and the like, film formation by the T-die method will suffice. Even in the case of the T-die method, however the intrinsic viscosity of the polybutylene terephthalate resin is preferably at least 0.5, and more preferably 0.7.

30 The inflation film-forming method is especially preferable since it produces a tubular film from which bags can be easily and inexpensively formed. Thus, bags can be formed readily by cutting the tubular film into tubular sections of selected length, followed by heat-sealing one end of each tubular section.

The fluid to be introduced into the packaging material of the present invention can be virtually any material that exhibits fluidity at its packaging temperature. Specific examples of materials that can be 35 packaged according to the present invention include materials that are normally in a liquid state such as natural fruit juice and cow's milk, and materials that are normally solid, but are in a liquid state at elevated packaging temperatures, such as curry roux, chocolates, molten resin materials such as hot-melt adhesives, and industrial materials, such as waxes and coating materials.

The polybutylene terephthalate resin packaging material according to the present invention is especially 40 characterized in that its "plastic" odour and taste are not transferred to foodstuffs with which it may come into contact due to polybutylene terephthalate being inherently free from a "plastic" odour and/or due to the fact that it does not absorb the fragrant components of its contents. Moreover, the foregoing properties are maintained even at high temperatures.

Bags or containers formed of polybutylene terephthalate may be packed with a water-based food or 45 drink, such as natural fruit juice or cow's milk, at a temperature of between about 80 to 100 °C (i.e., after high-temperature sterilization thereof), or with an oil-based food or drink such as curry roux or chocolate at a temperature of about 100 to 180 °C after high-temperature preparation). At such a high packaging temperature, conventional polyolefin containers undergo resin decomposition or dissolution and thereby intensely change the taste and fragrance of the food or drink contents. The packaging material of the 50 present invention, however, does not have such a problem.

Furthermore, films made of polybutylene terephthalate resin are heat-sealable, and exhibit excellent printability and mechanical strength properties. As such, polybutylene terephthalate films are particularly adapted for use in the form of a single layer food packaging bag.

Conventional films used as sealants exhibit not only poor inherent strength properties, but also poor 55 mechanical strength properties at the heat-sealed portions thereof. As a result, conventional packaging films are liable to be relatively weak along the heat-sealed portions to thereby cause bags formed of such films to rupture. Accordingly, the thickness of such conventional films must be increased to prevent bag rupturing.

In contrast, films made of polybutylene terephthalate resin not only exhibit high inherent properties, but also exhibit excellent mechanical strength properties at the heat-sealed portions thereof. As a result, bags formed of polybutylene terephthalate do not break at the heat-sealed portions, and are unlikely to rupture during transportation and handling. Accordingly, the thickness of the polybutylene terephthalate films can be reduced to achieve economic advantages. For example, an inflated film of polybutylene terephthalate resin exhibits tensile strength properties 2-3 times greater than inflated films of either low-density polyethylene or polypropylene, in addition to exhibiting superior tear strength and impact properties.

From the viewpoint of heat resistance alone, there are films that are superior to the films made of polybutylene terephthalate resin. However, such films are inadequate in terms of other necessary packaging performance characteristics, such as fragrance retentivity, and are typically more expensive than polybutylene terephthalate resins due to the higher prices associated with the cost of the resins themselves or due to the increased cost associated with film fabrication (i.e., because such resins cannot usually be formed into films using inflation film-forming techniques.) Furthermore, such films are typically used in a composite laminated form in combination with other films to provide heat sealability and thus enable the composite film to be formed into bags. As a result, the conventional composite films decrease productivity and present problems such as peeling at the lamination interface. In contrast, the films made of polybutylene terephthalate resin to be used in the present invention can be formed by inflation film forming techniques and exhibit excellent packaging characteristics even in a single-layer form. Thus, economical food packaging bags may be provided which can be filled at high temperatures with improved productivity.

The packaging material of the present invention can, in and of itself, be used in the form of a bag or a container, in addition to being used in the form of a liner bag for a metal or resin drum, for example.

As described above, the high-temperature polybutylene terephthalate packaging materials of the present invention exhibit excellent fragrance retentivity and are free from the peculiar "plastics" odour and taste associated with polyethylene and polypropylene resins. As a result, the polybutylene terephthalate packaging material does not affect the odour or taste of foods with which it comes into contact.

Moreover, these beneficial properties are present even at high temperatures. Therefore, polybutylene terephthalate films are particularly well suited for use as a high-temperature packaging material for foodstuffs and/or molten resin materials. Polybutylene terephthalate films can be formed by inflation film-forming techniques and are heat-sealable. Moreover, such polybutylene terephthalate films have excellent characteristics even in a single-layer form, so as to provide economical bags for packaging foods and industrial products.

Examples

The following non-limiting Examples will further illustrate the present invention.

Examples 1 and Comparative Example 1

A polybutylene terephthalate resin having an intrinsic viscosity of 1.4 and a melting point of 228° C was used to form a film by the inflation film-forming technique under the following conditions. Specifically, a tubular film was extruded with a 50mm extruder at a die lip space of 1mm and at a resin temperature of 260 to 265° C. The tubular film was drawn at a blow ratio of 1.5 and at a draw rate of 40./min to form a tubular film having a thickness of 50µm and a flat width of 95cm. The tubular film was cut into a piece having a length of 180cm, one end of which was then heat-sealed with an impulse sealer to produce a bag.

The polybutylene terephthalate bag thus produced was used as a liner bag for 200l high-density polyethylene drum to be packed with 180l of orange juice (reduced and condensed drink of 100% fruit juice) having a temperature of 85° C. The opening of the bag was closed with a nylon tie-lap band and covered with a high-density polyethylene lid, followed by storage at ambient temperatures. After one week, the bag was opened. The quantitative determination of a fragrant component (d-limonene) in the above-mentioned orange juice was made along with that of d-limonene absorbed on the liner bag and on the high-density polyethylene drum. Organoleptic tests were also conducted as to changes in the taste and smell of the orange juice and whether or not the smell of the orange juice was transferred to the liner bag and the drum. The results of evaluation were expressed in terms of the number of panelists who made evaluation of no change among 10 panelists.

In Comparative Example 1, substantially the same tests as those of Example 1 were conducted except that a low-density polyethylene bag having a thickness of 80 µm was used as the liner bag.

In the organoleptic tests, the changes in the taste and smell of the orange juice were examined by comparison of the extent of the changes using a case of orange juice packed in glass containers as a control.

The results are shown in Table 1.

Table 1

	Amt. of d-limonene				Taste, Smell		
	Orange juice before packing (ppm)	Orange juice after storage (ppm)	Liner bag (mg/cm ²)	Drum (mg/cm ²)	Orange juice after storage	Liner bag	Drum
Ex. 1	20	21*	0	0	10	8	10
Comp. Ex. 1	20	11.8	9.1x10 ⁻³	2.3x10 ⁻³	3	2	2

* The amount of d-dimonene in the juice slightly increased because d-limonene was formed through decomposition of juice components during storage.

Example 2 and Comparative Examples 2 to 4

As shown in Table 2, bags were respectively packed with various contents at various packing temperatures to examine the heat resistances thereof (changes in appearance including (1) deformation or melting and (2) change in hue). Organoleptic tests as to fragrance retentivity and changes in the taste and smell of the contents were conducted by 10 panelists.

The fragrance retentivity and the changes in the taste and smell of the contents were examined by comparison of the extents of changes with the case of the contents in the Pyrex container. The results of evaluation were expressed in terms of the number of panelists who made evaluation of no change among 10 panelists. The fragrance retentivity was evaluated by the smell inside a glass container having a bag packed with the contents, hermetically sealed by heat sealing and put into the container, which was then hermetically stoppered and allowed to stand at ordinary temperature for one week, according to three ratings: O (no recognizable smell), Δ (slightly recognizable smell) and X (strongly recognizable smell). The results are shown in Table 2.

Table 2

	Packaging material	Contents	Packing temp. (°C)	Heat resistance (change in appearance)	Fragrance retentivity	Changes of content	
						Taste	Smell
Ex. 2	Polybutylene terephthalate film bag 30 µm - thick 200 mm x 300 mm	Orange juice	85	not changed	0	9	9
		Cow's milk	85	"	0	10	10
		Sisalad oil	140	"	0	10	10
		Chocolate	180	"	0	10	10
Comp. Ex. 2	Low-density polyethylene film bag 60 µm - thick 200 mm x 300 mm	Hot-melt adhesive	160	"	0	-	10
		Cow's milk	50	not changed	Δ	4	3
		"	85	slightly deformed	X	3	1
		Sisalad oil	140	bag ruptured ; resin melted	-	0	0
Comp. Ex. 3	Polypropylene film bag 50 µm - thick 200 mm x 300 mm	Cow's milk	85	not changed	Δ	3	1
		Sisalad oil	140	largely deformed	Δ	2	3
		Chocolate	180	bag ruptured ; resin melted	-	1	2
		Cow's milk	50	not changed	Δ	4	7
Comp. Ex. 4	Ethylene-VAc film bag 50 µm - thick 200 mm x 300 mm	"	85	slightly deformed	X	3	3
		Chocolate	180	bag ruptured ; resin melted	-	0	1

While the invention has been described in connection with what is presently considered to be the most practical and preferred embodiment, it is to be understood that the invention is not to be limited to the disclosed embodiment, but on the contrary, is intended to cover various modifications and equivalent
5 arrangements included within the spirit and scope of the appended claims.

Claims

- 10 1. A method of packaging high-temperature materials comprising providing a material which is liquid at a packaging temperature of 80 ° C or greater, and then introducing the liquid material at the said packaging temperature into a container comprised of a polybutylene terephthalate resin.
2. A method as claimed in claim 1, wherein the said liquid material is liquid at a packaging temperature of 100 ° C or greater and is introduced into the said container at the said packaging temperature of 100 ° C or
15 greater.
3. A method as claimed in claim 1 or claim 2, wherein the said liquid material is an aqueous or oily food or beverage, a molten resin, a molten wax or a paint.
4. A method as claimed in any preceding claim, wherein the container is in the form of a bag.
5. A method as claimed in claim 4, wherein the polybutylene terephthalate has an intrinsic viscosity of at
20 least 1.0 and is formed by the inflation method into a film for fabricating the bag.
6. A method as claimed in any one of claims 1 to 3, wherein the container is in the form of a molded container.
7. A package comprising a liquid material at a temperature of 80 ° C or greater contained within a container which is formed of a polybutylene terephthalate resin.
- 25 8. A package as claimed in claim 7, wherein the said material is liquid at a packaging temperature of 100 ° C or greater and is introduced into the said container at the said packaging temperature of 100 ° C or greater.
9. A package as claimed in claim 7 or claim 8, wherein the liquid material is an aqueous or oily food or beverage, a molten resin, a molten wax or a paint.
10. A package as claimed in any of claims 7 to 9, wherein the container is in the form of a bag.
- 30 11. A package as claimed in claim 10, wherein the polybutylene terephthalate has an intrinsic viscosity of at least 1.0 and is formed by the inflation method into a film for fabricating the bag.
12. A package as claimed in any one of claims 7 to 9 wherein the container is in the form of a molded container.

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EUROPEAN SEARCH REPORT

Application Number

EP 90 30 8352

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
Y	EP-A-0 323 128 (POLYPLASTICS) * Page 4, lines 1-15; page 4, lines 44-54 *	1,3,4,5,6, 7,9,10,11, 12	B 65 B 63/08 B 65 B 25/00 B 65 D 65/38
Y	FR-A-1 388 199 (DOW CHEMICAL) * Page 5, column II, lines 7-47 *	1,3,4,5,6, 7,9,10,11, 12	
A	JP-A-6 222 131 (HOUSE SHOKUHIN) * Abstract *	1	
A	US-A-4 869 864 (POLYPLASTICS) * Column 2, lines 4-41 *	1,5,11	
A	EP-A-0 056 323 (DRG) * Page 1, line 1 - page 2, line 17 *	1	
A	GB-A-1 405 769 (BASF) * Claims *	1	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			B 65 B B 65 D
The present search report has been drawn up for all claims			
Place of search		Date of completion of search	Examiner
The Hague		30 October 90	JAGUSIAK A.H.G.
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			