



Office de la Propriété
Intellectuelle
du Canada

Un organisme
d'Industrie Canada

Canadian
Intellectual Property
Office

An agency of
Industry Canada

CA 2162008 C 2008/01/22

(11)(21) **2 162 008**

(12) **BREVET CANADIEN
CANADIAN PATENT**

(13) **C**

(22) Date de dépôt/Filing Date: 1995/11/02

(41) Mise à la disp. pub./Open to Public Insp.: 1996/05/04

(45) Date de délivrance/Issue Date: 2008/01/22

(30) Priorité/Priority: 1994/11/03 (DEP 44 39 248.6)

(51) Cl.Int./Int.Cl. *C08L 23/06* (2006.01),
B29C 47/00 (2006.01), *C08L 23/02* (2006.01),
C08L 23/30 (2006.01)

(72) Inventeurs/Inventors:
BREUERS, WERNER, DE;
BERTHOLD, JOACHIM, DE

(73) Propriétaire/Owner:
BASELL POLYOLEFINE GMBH, DE

(74) Agent: BERESKIN & PARR

(54) Titre : COMPOSITION POLYOLEFINIQUE POUR MOULAGE, MOINS APTE A PELER

(54) Title: POLYOLEFIN MOLDING COMPOSITION WITH A REDUCED TENDENCY TO PEEL

(57) Abrégé/Abstract:

A polyolefin molding composition which "peels" relatively little or not at all during extrusion comprises an olefin polymer and 0.001 to 5 phr of an oxidized wax which is an oxidized synthetic or semi-synthetic natural wax or an oxidized polyolefin wax.



ABSTRACT

Polyolefin molding composition with a reduced tendency to peel

5 A polyolefin molding composition which "peels" relatively little or not at all during extrusion comprises an olefin polymer and 0.001 to 5 phr of an oxidized wax which is an oxidized synthetic or semi-synthetic natural wax or an oxidized polyolefin wax.

DESCRIPTION

Polyolefin molding composition with a reduced tendency to peel

5 The invention relates to a polyolefin molding composition which "peels" relatively little or not at all during extrusion.

10 After preparation in the form of granules or grit, olefin polymers are processed to a finished component (product) by the extrusion or injection molding process. This finished component can be a film, a tube or a large or small hollow body. The problem of "peeling" occurs specifically during the processing of higher molecular weight polyolefins to give large hollow bodies (drums, tanks and the like).

15 "Peeling" is understood as meaning the phenomenon of filaments of the same material forming on the inside or outside of the plastic tube and later large hollow body on delivery of the polymer melt. These filaments interfere with the use of the vessel as intended, since, for
20 example, they can become detached from an inner surface of the drum and contaminate the product contained therein.

To eliminate peeling, it is customary to change the molecular weight distribution of the polymer or to adapt
25 the processing machines to suit a given product, for example by changing the nozzle geometry.

A change in the molecular weight distribution is in general undesirable, since it leads to a change in the product properties. Modification of the processing
30 machines is likewise usually not possible, since products from different manufacturers are used and the machines cannot be optimized for a certain product.

The object of the present invention was to prevent the occurrence of the "peeling effect" during processing of polyolefins without a change having to be made to the molecular weight distribution of the polymer or to the processing machines.

5

This object was achieved by addition of an oxidized wax during granulation of the polyolefin powder.

The invention thus relates to a polyolefin molding composition with a reduced
10 tendency to peel, comprising an olefin polymer and 0.001 to 5 phr of an oxidized wax.

The invention furthermore relates to a process for reducing peeling during
extrusion of polyolefin molding compositions. This process includes adding
15 0.001 to 5phr of an oxidized wax to the molding composition comprising an olefin polymer.

The polyolefin molding composition according to the invention comprises an olefin polymer, for example one of the following:

20

1. Polymers of mono- and diolefins, for example polyethylene of high, medium or low density (which can be optionally cross-linked), polypropylene, polysiobutylene, polybut-1-ene, polymethylpent-1-ene, polyisoprene or polybutadiene, and polymers of cycloolefins, such as, for example,
25 cyclopentene or norbornene.

2. Mixtures of the polymers mentioned under 1), for example mixtures of polypropylene with polyethylene or with polyisobutylene.

30 3. Copolymers of mono- and diolefins with one another or with other vinyl monomers, such as, for example, ethylene/propylene copolymers, propylene/but-1-ene copolymers, propylene/isobutylene copolymers, ethylene/but-1-ene copolymers, propylene/butadiene copolymers, isobutylene/isoprene copolymers,

ethylene/alkyl acrylate copolymers, ethylene/alkyl methacrylate copolymers, ethylene/vinyl acetate copolymers or ethylene/acrylic acid copolymers and salts thereof (ionomers), as well as terpolymers of ethylene
5 with propylene and a diene, such as hexadiene, dicyclopentadiene or ethylidenenorbornene.

Polyethylene, polypropylene, polybut-1-ene, polyhex-1-ene, poly-4-methylenepent-1-ene, polyoct-1-ene or copolymers thereof are preferred.

10 In addition to the olefin polymer, the molding composition comprises waxes. These waxes are oxidized fully synthetic or semi-synthetic waxes or polyolefin waxes. Preferred waxes are oxidized semi-synthetic waxes based on crude montan wax and polyolefin waxes.

15 The preferred oxidized semi-synthetic wax based on crude montan wax has the following characteristic data:

drop point	79 to 90°C
acid number	115 to 155 mg of KOH/g
hydrolysis number	140 to 175 mg of KOH/g
20 density	0.97 to 1.03 g/cm ³

The preferred oxidized wax based on polyolefin wax has the following characteristic data:

drop point	95 to 130°C
acid number	15 to 70 mg of KOH/g
25 hydrolysis number	25 to 115 mg of KOH/g
density	0.90 to 1.03 g/cm ³

The preferred oxidized semi-synthetic wax based on crude montan wax is formed by oxidative bleaching of crude montan wax. Waxes of this type have been commercially
30 obtainable for many years.

The preferred oxidized wax based on polyolefin wax is formed by bulk polymerization in the Ziegler low pressure process. The resulting polyolefin wax is converted into

oxidized products by treatment of the melt with air. These waxes have also been commercially obtainable for many years.

The oxidized wax is added to the polymer in a concentration of 0.001 to 5 phr (parts per 100 parts of resin), preferably 0.01 to 0.5 phr. In addition, the molding composition according to the invention can also comprise further additives, such as, for example, antioxidants, for example alkylated monophenols, alkylated hydroquinones, hydroxylated diphenyl thioethers, alkylidenebisphenols, benzyl compounds, acylaminophenols, esters of β -(5-t-butyl-4-hydroxy-3-methylphenyl)propionic acid, amides of β -(3,5-di-t-butyl-4-hydroxyphenyl)propionic acid, UV absorbers and light stabilizers, for example 2-(2'-hydroxyphenyl)benzotriazoles, 2-hydroxybenzophenones, oxalic diamides, esters of unsubstituted or substituted benzoic acids, acrylates, nickel compounds, sterically hindered amines (HALS), metal deactivators, phosphates and phosphites, peroxide-destroying compounds, basic costabilizers, nucleating agents, fillers and reinforcing materials, plasticizers, optical brighteners, flameproofing agents, antistatics, blowing agents, lubricants and pigments.

The oxidized waxes to be used according to the invention and, if appropriate, the other additives are incorporated by the methods customary for compounding polyolefins. They are preferably added during the granulation.

The polyolefin molding composition according to the invention results in finished components which no longer show a peeling effect.

The following examples are intended to illustrate the invention. The "peeling effect" is characterized in the following manner. Drums having a capacity of 200 l are produced from a polyolefin molding composition according to the invention at different throughputs on an extrusion

blow molding machine. The drums are then assessed visually with a classification into a "peel rating" of between 1 and 5. The rating 1 means that no filaments at all are to be observed on the drum surface. The drum surface is smooth and intact. Classification into ratings 2-5 is made according to the frequency of the occurrence of polymer filaments on the drum surface and the thickness thereof.

Example 1

10 A polyethylene having an MFR 190/21.6 of 2.0 g/10 minutes and a density of 0.950 g/cm³ was mixed in a mixing container with 0.12 phr of pentaerythritol tetrakis[3-(3-,5-di-t-butyl-4-hydroxyphenyl)propionate] and 0.2 phr of an oxidized polyethylene wax (density 0.97 - 0.99 g/cm³, AN
15 15 - 17 mg of KOH/g, HN 20 - 35 mg of KOH/g) and the mixture was granulated at 290°C by means of an extruder. 400 kg of these granules were processed to give 200 l drums on an extrusion blow molding line. The drums thus obtained had a smooth inner and outer surface. The "peel
20 rating" was 1.

Example 2

A polyethylene having an MFR 190/21.6 of 2.0 g/10 minutes and a density of 0.950 g/cm³ was mixed in a mixing container with 0.12 phr of pentaerythritol tetrakis[3-(3-,5-di-t-butyl-4-hydroxyphenyl)propionate] and 0.2 phr of an
25 oxidized polyethylene wax (density 0.97 - 0.99 g/cm³, AN 15 - 17 mg of KOH/g, HN 20 - 35 mg of KOH/g) and the mixture was granulated together with 0.8 phr of a blue batch (masterbatch with blue pigment) at 290°C by means
30 of an extruder. 400 kg of these blue granules were processed to give 200 l drums on an extrusion blow molding line. The blue drums thus obtained had a smooth inner and outer surface. The "peel rating" was 1.

Comparison example A

The same polyethylene as in Examples 1 and 2 was stabilized in a mixing container with 0.12 phr of pentaerythritol tetrakis[3-(3-,5-di-t-butyl-4-
5 hydroxyphenyl)propionate] and the mixture was granulated at 290°C by means of an extruder. 400 kg of these granules were extruded to give 200 l drums on an extrusion blow molding line. The drums thus obtained had a "peeling effect" of rating 3, 4 or 5 on the inner or
10 outer surface.

Comparison example B

The same polyethylene as in Examples 1 and 2 was mixed in a mixing container with 0.12 phr of pentaerythritol-tetrakis[3-(3-,5-di-t-butyl-4-hydroxyphenyl)propionate]
15 and together with 0.8 phr of a blue batch (masterbatch with blue pigment), and the mixture was granulated at 290°C by means of an extruder. 400 kg of these blue granules were extruded to give 200 l drums on an extrusion blow molding unit. The blue drums thus obtained
20 had a "peeling effect" of rating 3, 4 or 5 on the inner or outer surface.

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. A process for reducing peeling during extrusion of a polyolefin molding
5 composition, which comprises adding 0.001 to 5 phr of an oxidized wax to the molding composition comprising an olefin polymer.
2. The process as claimed in claim 1, wherein the oxidized wax is an oxidized
10 polyolefin wax.
3. The process as claimed in claim 1, wherein the oxidized wax is an oxidized
fully synthetic wax or an oxidized semi-synthetic wax.
4. The process as claimed in claim 1, wherein the olefin polymer is
15 polyethylene, polypropylene, polybut-1-ene, polyhex-1-ene, poly-4-methylenepent-1-ene, polyoct-1-ene or a copolymer thereof.
5. The process as claimed in claim 4, wherein the olefin polymer is
20 polyethylene.
6. The process as claimed in claim 1, wherein drums are produced during
extrusion.

25

30

35

40