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(54) Titre : COMPOSITION POLYMERE DESTINEE A DES CAPSULES DE BOUTEILLES A VIS
(54) Title: POLYMER COMPOSITION FOR BOTTLE SCREW CAPS

(57) **Abrégé/Abstract:**

The present invention relates to a polymer composition, especially used for manufacturing screw caps intended for drinks bottles, which are easy to unscrew and at the same time avoid transmitting a taste and/or a smell to the drinks contained in the bottles. The composition essentially comprises (1) a polymer, except a polymer based on a conjugated diene, (2) at least one stabilizer and (3) at least one additive in an amount from 0.01 to 1% by weight, chosen from (a) natural, modified or refined, lignite waxes or montan waxes, (b) polyolefin-based or paraffin-based polar waxes, and (c) refractory or metal oxides grafted by polar organic compounds.

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(54) Title: POLYMER COMPOSITION FOR BOTTLE SCREW CAPS (57) Abstract The present invention relates to a polymer composition, especially used for manufacturing screw caps intended for drinks bottles, which are easy to unscrew and at the same time avoid transmitting a taste and/or a smell to the drinks contained in the bottles. The composition essentially comprises (1) a polymer, except a polymer based on a conjugated diene, (2) at least one stabilizer and (3) at least one additive in an amount from 0.01 to 1% by weight, chosen from (a) natural, modified or refined, lignite waxes or montan waxes, (b) polyolefin-based or paraffin-based polar waxes, and (c) refractory or metal oxides grafted by polar organic compounds.		

POLYMER COMPOSITION FOR BOTTLE SCREW CAPS

The present invention relates to a polymer composition and its use for the manufacture of screw caps for bottles containing, in particular, drinks such as soda and carbonated or uncarbonated, mineral or natural, water.

5 The technical problem encountered in the use of plastic screw caps intended for bottles containing drinks is often related to the difficulty that the consumer has in unscrewing the cap from the bottle and to the unpleasant taste and unpleasant smell that the cap may give to the drink contained in the bottle. It is known to use a polyethylene composition containing an "amide wax" which is, for example, the product of the reaction of a fatty acid with ammonia, an amine or a diamine. Screw caps manufactured
10 with such a composition are easy to unscrew from the bottle but have the drawback of being able to transfer a taste and/or a smell to the contents of the bottle.

The WO Patent No 95/00599 describes a liner for reclosable container closure containing a lubricating agent such as silicones, fatty acid amines e.g. ethylene bis(stearamide), oleamide and erucamide, metal salts of fatty acids and inorganic
15 materials such as talc, mica, fumed silica and calcium silicate.

The US Patent No 4,256,234 describes a container closure having an easy openable liner. More particularly it describes the liner and the olefin resin composition of said liner that contains a conjugated diene polymer. The disclosed composition is used for a liner and not for a plastic screw caps as in the present invention.

20 The US Patent No 5,834,079 describes a film having at least one layer containing a zeolite and an oxygen scavenger. The film layers disclosed in the Patent does not comprise all the components of the present invention composition.

The US Patent No 3,923,198 describes a container that is hermetically sealed

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with an easy opening tape, at least a portion of which opacifies when stressed, providing a visual indication that the closure has been tampered with. The components of the present invention composition are used in different proportions.

A polymer composition used for screw caps for bottles containing drinks such as sodas and carbonated or uncarbonated, mineral or natural, water, which are easy to unscrew and at the same time avoid a taste and/or a smell being transferred to the drinks contained in the bottles, has been found.

The composition essentially comprises

- (1) a polymer except a polymer based on a conjugated diene and
- 10 (2) at least one stabilizer chosen from neutralizing agents, short-term antioxidants, long-term antioxidants and UV stabilizers, and
- (3) at least one additive in an amount from 0.01 to 1%, preferably from 0.1 to 0.4%, by weight chosen from
 - (a) natural, modified or refined lignite waxes or montan waxes,
 - 15 (b) polyolefin-based or paraffin-based polar waxes, and
 - (c) refractory or metal oxides grafted by polar organic compounds, especially polyalkylene glycol compounds.

The polymer may be an ethylene (co)polymer, a propylene (co)polymer or a polyethylene terephthalate (PET), preferably a homopolyethylene or a copolymer of
 20 ethylene with at least one C₃ to C₁₀ alpha-olefin. The ethylene (co)polymer may have a density ranging from 0.930 to 0.965, preferably from 0.948 to 0.955 g/cm³. It may be a copolymer of ethylene with at least one C₃ to C₁₀ alpha-olefin, especially propylene, 1-butene, 1-hexene, 4-methyl-1-pentene or 1-octene. The polyethylene may have a melt flow index (measured according to the ASTM-D-1238, condition E, standard) ranging
 25 from 0.5 to 30, preferably from 1 to 12 g/10 minutes. It may also have a molecular mass distribution width (expressed by the ratio of the weight-average molecular mass, M_w, to the number-average molecular mass, M_n) ranging from 1.5 to 6, preferably from 2 to 5.

The composition comprises at least one stabilizer chosen from neutralizing agents, short-term antioxidants, long-term antioxidants and UV stabilizers, preferably in
 30 an amount ranging from 0.02 to 0.5% by weight.

The neutralizing agent may be chosen from zinc stearate, calcium stearate and zinc oxide, in an amount ranging, for example, from 0 to 0.5%, preferably from 0.05 to 0.5%, by weight.

The short-term antioxidant (also called a secondary antioxidant or so-called "process" antioxidant) may be chosen from phosphorus derivatives such as organophosphorus compounds, especially phosphites, in an amount ranging, for example, from 0 to 0.2%, preferably from 0.02 to 0.2%, by weight. The products sold
5 under the brand name "IRGAFOS 168"® or "IRGAFOS P-EPQ"® manufactured by Ciba Additives may be used.

The long-term antioxidant (also called the primary antioxidant) may be chosen from sterically hindered phenolic compounds and thioesters, such as "IRGANOX 1010"®, "IRGANOX 1076"® or "IRGANOX PS802"® manufactured by Ciba
10 Additives, in an amount ranging, for example, from 0 to 0.2%, preferably from 0.02 to 0.2%, by weight.

The UV stabilizer may be chosen from hindered amines, such as "TINUVIN 622"® manufactured by Ciba Additives, in an amount ranging, for example, from 0 to 0.2%, preferably from 0.02 to 0.2%, by weight.

15 The composition according to the invention comprises at least one additive which, because of its presence, allows a cap manufactured from the said composition and screwed onto a bottle to be easily unscrewed and which, at the same time, does not transfer significant taste or smell to the drink contained in the bottle.

The additive may be a lignite wax or a montan wax which, in the natural state, is
20 essentially a mixture of C₁₆ to C₃₄, preferably C₂₂ to C₃₂, monocarboxylic or dicarboxylic or hydroxycarboxylic acids, of esters of these acids, of C₁₈ to C₃₄, preferably C₂₂ to C₃₂, alcohols and of C₂₅ to C₃₃ aliphatic hydrocarbons. The lignite wax or montan wax may be natural wax or preferably refined wax, that is to say the wax after it has undergone a resin extraction operation (also called deresinification) and/or a bleaching or oxidizing-
25 decoloration operation and/or a saponification operation. As regards montan wax, that sold under the brand name "E Wax"® or "OP Wax"® manufactured by Clariant may be used.

The additive may be a polyolefin-based or paraffin-based polar wax, especially chosen from oxidized polyolefin waxes or Fischer-Tropsch waxes, for example polar
30 waxes of homopolymers or copolymers of a C₂ to C₁₀ olefin, for example ethylene, propylene or butene, or of a C₅ to C₁₀ higher alpha-olefin. Polar waxes are generally oxidized waxes which may be obtained by oxidation reactions in the presence of air or oxygen, or by grafting reactions, for example, maleic anhydride grafting reactions. As a

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polar polyolefin wax, that sold under the brand name "PED 191 Wax"® manufactured by Hoechst may be used.

The additive may be a refractory or metal oxide grafted by polar organic compounds. The refractory or metal oxide may be a magnesium oxide, a zinc oxide, a titanium oxide, a silica, an alumina, an aluminosilicate or a mixture of these oxides. The polar organic compound grafted to the refractory or metal oxide may be any organic compound having one or more oxygen atoms, sulphur atoms, nitrogen atoms, phosphorus atoms and, optionally, halogen, such as fluorine, chlorine, bromine or iodine, atoms. The grafted polar organic compound may preferably be a polyalkylene glycol, such as a polyethylene glycol or a polypropylene glycol, or an ethylene/propylene glycol copolymer. In particular, it is preferred to use a polyethylene glycol. In particular, a silica which is grafted, for example, by a polyethylene glycol and which consists of particles having an average diameter (measured by a "Malvern Mastersizer" with a focal length of 45 mm) from 1 to 10 μm may be used. The amount of grafted polar organic compound in the refractory or metal oxide may be from 0.1 to 25% by weight. More especially, a silica grafted by a polyethylene glycol, under the brand name "SYLOBLOC K550"® sold by Grace may be used.

The composition may be prepared according to a method in which at least one of the additives and at least one of the stabilizers are added to the polymer in the solid state or in the melt state and are blended with the latter in the solid state or in the melt state. It may be prepared in particular by blending the various constituents together, preferably in an extruder or granulator capable of producing the blend at a temperature above the melting point of the polymer. In this case, the composition is obtained in the form of granules.

The composition may in particular be prepared by blending the polymer with a masterbatch which is a concentrated composition of at least one of the aforementioned stabilizers and of at least one of the aforementioned additives in a polymer like that mentioned above. The composition of the "masterbatch" essentially comprises the same constituents as those of the composition, that is to say:

- (1) a polymer, except a polymer based on a conjugated diene,
- (2) at least one stabilizer chosen from

(a) neutralizing agents, for example in an amount ranging from 0 to 30%, preferably from 2 to 30%, by weight,

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- (b) short-term antioxidants, for example in an amount ranging from 0 to 15%, preferably from 1 to 15%, by weight,
- (c) long-term antioxidants, for example in an amount ranging from 0 to 15%, preferably from 1 to 15%, by weight,
- 5 (d) UV stabilizers, for example in an amount ranging from 0 to 15%, preferably from 1 to 15%, by weight, and
- (3) at least one additive in an amount from more than 5 to 25%, by weight, chosen from
 - (a) natural, modified or refined lignite waxes or montan waxes,
 - 10 (b) polyolefin-based or paraffin-based polar waxes, and
 - (c) refractory or metal oxides grafted by polar organic compounds, especially polyalkylene glycol compounds.

In this case, the composition may be prepared using a method in which the masterbatch is added to the polymer in the solid state or in the melt state and is blended with the
 15 latter in the solid state or in the melt state.

The composition may also be prepared by blending the polymer with a preblend containing at least one of the stabilizers and at least one of the additives, such as those mentioned above. The composition of the said blend essentially comprises the same constituents as those of the composition, apart from the polymer, that is to say for a total
 20 of 100 parts by weight:

- (1) from more than 5 to less than 95 parts, preferably from 10 to 90 parts, by weight of at least one stabilizer chosen from
 - (a) neutralizing agents,
 - (b) short-term antioxidants,
 - 25 (c) long-term antioxidants,
 - (d) UV stabilizers and
- (2) from more than 5 to less than 95 parts, preferably from 10 to 90 parts, by weight of at least one additive chosen from
 - (a) natural, modified or refined lignite waxes or montan waxes,
 - 30 (b) polyolefin-based or paraffin-based polar waxes, and
 - (c) refractory or metal oxides grafted by polar organic compounds, especially polyalkylene glycol compounds.

In this case, the composition may be prepared according to a method in which the

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preblend is added to the polymer in the solid state or in the melt state and is blended with the latter in the solid state or in the melt state.

The invention also relates to the use of the composition, of the masterbatch or of the preblend in the manufacture of caps intended to be screwed onto plastic bottles containing, in particular, drinks such as sodas and carbonated or uncarbonated, mineral or natural, water.

The invention also relates to the screw caps used for bottles containing drinks and consisting of the composition described above.

The screw caps may be manufactured by an operation of injection-moulding or compression-moulding the composition described above and used either in the form of a blend or in the form of granules. The operation essentially consists in injection-moulding or compression-moulding the composition at a temperature of 170 to 250°C, preferably 200 to 240°C.

Examples

The following non-limiting examples illustrate the present invention.

In the examples, the compositions are manufactured from an ethylene/1-butene copolymer in powder form, having a density of 951 kg/m³ and a melt flow index (measured according to the ASTM-D-1238, condition E, standard) of 2 g/10 minutes, or else a density of 952 kg/m³ and a melt flow index (measured according to the ASTM-D-1238, condition E, standard) of 11 g/10 minutes. The compositions are extruded at 200°C using an extruder of the "Werner 53"®

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brand. The caps are manufactured by an operation of injection-moulding the composition at a temperature of 200°C.

In the following examples, the cap unscrewing torques on PET preforms are measured with an apparatus known
5 by the brand name "Ernst Torque Tester"® manufactured by "Verpackungen A.G.". The caps are screwed on beforehand with a torque of 2.6 Nm. The unscrewing test is carried out after conditioning the caps for one week at 23°C.

In the examples, the taste tests are carried out
10 by a human panel of water tasters (non-smokers) on caps manufactured using the composition. The caps, after prior aeration for half an hour at room temperature, are immersed in a natural mineral water in a clean bottle placed in an oven at 60°C. Next, the water is recovered and diluted
15 twice, four times, eight times and sixteen times by volume with respect to the initial solution using the natural mineral water. Each solution is assigned a rating from 0 to 5, depending

on the taste transferred to the water by the cap, the lowest rating being assigned in the case of a substantially non-existent taste.

In the examples, the smell tests are carried out by the same human panel on caps manufactured using the compositions. These caps, after prior aeration for half an hour at room temperature, are put into clean bottles placed in an oven at 80°C. A control specimen, made of additive-free polyethylene and having a smell rating of 0.5, undergoes the same preparation treatment. Next, each specimen is compared with the control and a rating is assigned between 0 (no smell) and 2 (strong smell).

10 **Example No. 1: the additive is a polar wax**

	Comparative composition	Comparative composition based on amide wax	Composition according to the invention
Polymer	ethylene/1-butene copolymer density: 951 kg/m ³ MFI(*): 2 g/10 minutes		
Stabilizers (% by weight)	calcium stearate: 0.15% "IRGAFOS 168"®: 0.1% "TINUVIN 622"®: 0.05%		
Additive (% by weight)		"UNISLIP 1759"® from Unichema: 0.2%	"PED 191 Wax"®: 0.2% (**)
Unscrewing torque (inch.lb)	17	15	15
Taste (rating from 0 to 5)	1.2	2.5	1.4
Smell (rating from 0 to 2)	0.4	0.8	0.4

(*) Measured according to the ASTM-D-1238, condition E, standard.

(**) "PED 191 Wax"® is a polar polyolefin wax.

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Example No. 2: the additive is a polar wax

	Comparative composition	Comparative composition based on amide wax	Composition according to the invention
Polymer	ethylene/1-butene copolymer density: 951 kg/m ³ MFI(*): 2 g/10 minutes		
Stabilizers (% by weight)	zinc stearate: 0.15% "IRGAFOS 168"®: 0.08% "IRGANOX 1010"®: 0.06%		
Additive (% by weight)		"UNISLIP 1759"® from Unichema: 0.2%	"OP Wax"®: 0.2% (**)
Unscrewing torque (inch.lb)	17	15	15
Taste (rating from 0 to 5)	1.2	2.5	1.5
Smell (rating from 0 to 2)	0.4	0.8	0.5

(*) Measured according to the ASTM-D-1238 standard.

5 (**) "OP Wax"® is a montan wax.

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Example No. 3: the additive is a grafted silica

	Comparative composition	Comparative composition based on amide wax	Composition according to the invention
Polymer	ethylene/1-butene copolymer density: 952 kg/m ³ MFI(*): 11 g/10 minutes		
Stabilizers (% by weight)	calcium stearate: 0.15% "IRGAFOS 168"®: 0.06% "IRGANOX 1010"®: 0.04% "TINUVIN 622"®: 0.05%		
Additive (% by weight)		"UNISLIP 1759"® from Unichema: 0.2%	"SYLOBLOC K550"®: 0.3% (**)
Unscrewing torque (inch.lb)	17	15	15.5
Taste (rating from 0 to 5)	1	2.5	1.5
Smell (rating from 0 to 2)	0.4	0.8	0.5

(*) Measured according to the ASTM-D-1238 standard.

5 (**) "SYLOBLOC K550"® is a silica grafted by a polyethylene glycol.

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CLAIMS:

1. A polymer composition used for manufacturing screw caps intended for drinks bottles, with the proviso that said polymer composition does not include a polymer based on a
5 conjugated diene, essentially comprising:

(1) a polymer, and

(2) at least one stabilizer chosen from a neutralizing agent, a short-term antioxidant, a long-term antioxidant and a UV stabilizer,

10 and further comprising at least one additive in an amount from 0.01 to 1% by weight chosen from:

(a) natural, modified or refined lignite waxes or montan waxes,

(b) polyolefin-based or paraffin-based polar waxes, and

15 (c) refractory or metal oxides grafted by polyalkylene glycols.

2. The composition according to claim 1, wherein the polymer is an ethylene (co)polymer, a propylene (co)polymer or a polyethylene terephthalate.

20 3. The composition according to claim 2, wherein the ethylene (co)polymer is a homopolyethylene or a copolymer of ethylene with at least one C₃ to C₁₀ alpha-olefin.

4. The composition according to claim 3, wherein the ethylene (co)polymer has a density of 0.930 to 0.965 g/cm³
25 and a melt flow index (measured according to the ASTM-D-1238, condition E, standard) of 0.5 to 30 g/10 minutes.

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5. The composition according to any one of claims 1 to 4, wherein at least one of the stabilizers is present in an amount from 0.02 to 0.5% by weight.

6. The composition according to any one of claims 1 to 5, wherein the neutralizing agent is chosen from zinc stearate, calcium stearate and zinc oxide, and is present in an amount up to 0.5% by weight.

7. The composition according to any one of claims 1 to 6, wherein the short-term antioxidant is a phosphorus derivative and is present in an amount up to 0.2% by weight.

8. The composition according to any one of claims 1 to 7, wherein the short-term antioxidant is a phosphorus derivative chosen from organophosphorus compounds.

9. The composition according to any one of claims 1 to 8, wherein the short-term antioxidant is chosen from phosphites.

10. The composition according to any one of claims 1 to 9, wherein the long-term antioxidant is chosen from sterically hindered phenolic compounds and thioesters and is present in an amount up to 0.2% by weight.

11. The composition according to any one of claims 1 to 10, wherein the UV stabilizer is a hindered amine and is present in an amount up to 0.2% by weight.

12. The composition according to any one of claims 1 to 11, wherein the at least one additive is a lignite wax or a montan wax which, in the natural state, essentially consists of C₁₆ to C₃₄ monocarboxylic or dicarboxylic or hydroxycarboxylic acids, or esters of these acids, or C₁₈ to C₃₄ alcohols or C₂₅ to C₃₃ aliphatic hydrocarbons.

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13. The composition according to any one of claims 1 to 11, wherein the at least one additive is a polar wax chosen from oxidized polyolefin waxes and oxidized Fischer-Tropsch waxes.

5 14. The composition according to any one of claims 1 to 11 and 13, wherein the at least one additive is a polar wax of a C₂ to C₁₀ olefin homopolymer or copolymer.

15. The composition according to any one of claims 1 to 11, wherein the at least one additive is a refractory or
10 metal oxide grafted by polyalkylene glycols, in which at least one additive the refractory or metal oxide is chosen from magnesium oxide, zinc oxide, titanium oxide, a silica, an alumina, an aluminosilicate and a mixture of these oxides.

15 16. The composition according to any one of claims 1 to 11 and 15, wherein the at least one additive is a refractory or metal oxide grafted by a polyethylene glycol.

17. A polymer masterbatch used in the manufacture of screw caps intended for drinks bottles, with the proviso
20 that said polymer masterbatch does not include a polymer based on a conjugated diene, essentially comprising:

(1) a polymer,

(2) at least one stabilizer chosen from a neutralizing agent, a short-term antioxidant, a long-term antioxidant and
25 a UV stabilizer and

(3) at least one additive in an amount from more than 5 to 25%, by weight, chosen from:

(a) natural, modified or refined lignite waxes or montan waxes,

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(b) polyolefin-based or paraffin-based polar waxes, and

(c) refractory or metal oxides grafted by polyalkylene glycols.

18. A preblend used in the manufacture of screw caps
5 intended for drinks bottles, comprising, for a total of 100 parts by weight:

(1) from more than 5 to less than 95 parts by weight of at least one stabilizer chosen from a neutralizing agent, a short-term antioxidant, a long-term antioxidant and a UV
10 stabilizer, and

(2) from more than 5 to less than 95 parts by weight of at least one additive chosen from:

(a) natural, modified or refined lignite waxes or montan waxes,

15 (b) polyolefin-based or paraffin-based polar waxes, and

(c) refractory or metal oxides grafted by polyalkylene glycols.

19. A process for preparing the composition according to any one of claims 1 to 16, wherein at least one of the
20 additives and at least one of the stabilizers are added to the polymer in the solid state or in the melt state and are blended with the latter in the solid state or in the melt state.

20. A process for preparing the composition according
25 to any one of claims 1 to 16, wherein the masterbatch according to claim 17 is added to the rest of the polymer in the solid state or in the melt state and is blended with the latter in the solid state or in the melt state.

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21. A process for preparing the composition according to any one of claims 1 to 16, wherein the preblend according to claim 18 is added to the polymer in the solid state or in the melt state and is blended with the latter in the solid
5 state or in the melt state.

22. Use of the composition, of the masterbatch or of the preblend according to any one of claims 1 to 18 for manufacturing screw caps intended for drinks bottles.

23. Screw caps used for bottles containing drinks and
10 consisting of the composition according to any one of claims 1 to 16.

24. A process for manufacturing screw caps according to claim 23, wherein an operation of injection-moulding or compression-moulding the composition is carried out at a
15 temperature from 170 to 250°C.

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