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(11)

EP 3 753 435 A1

(12)

EUROPEAN PATENT APPLICATION
published in accordance with Art. 153(4) EPC

(43) Date of publication:

23.12.2020 Bulletin 2020/52

(51) Int Cl.:

A41G 3/00 ^(2006.01) **D06M 13/11** ^(2006.01)
D06M 13/12 ^(2006.01) **D06M 13/152** ^(2006.01)

(21) Application number: **19754968.6**

(86) International application number:

PCT/JP2019/004787

(22) Date of filing: **12.02.2019**

(87) International publication number:

WO 2019/159866 (22.08.2019 Gazette 2019/34)

(84) Designated Contracting States:

**AL AT BE BG CH CY CZ DE DK EE ES FI FR GB
GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO
PL PT RO RS SE SI SK SM TR**

Designated Extension States:

BA ME

Designated Validation States:

KH MA MD TN

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(30) Priority: **15.02.2018 JP 2018024955**

(54) **HUMAN HAIR FIBER TREATMENT AGENT**

(57) A human hair fiber treatment agent comprised of one or more compositions for treating human hair fibers having free ends at both ends, wherein the human hair fiber treatment agent comprises the following components (A) to (C) in the formulation thereof: (A): at least one formaldehyde derivative selected from the group consisting of formaldehyde, and the like; (B): phenol com-

pound(s) having electron-donating group(s) on at least one of the meta positions and having hydrogen atom(s) on at least one of the ortho positions and the para position, with the proviso that the electron-donating group at the meta position optionally forms, together with a carbon atom attached thereto, a benzene ring optionally substituted with hydroxy group(s); and (C): water.

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Description

Field of the Invention

5 **[0001]** The present invention relates to a human hair fiber treatment agent which semi-permanently or permanently changes the shape of human hair fibers as an industrial material.

Background of the Invention

10 **[0002]** Heretofore, synthetic hair fibers and human hair fibers have been widely known as fibers for hair implantation for making wigs.

[0003] Wigs using synthetic hair provide the following advantages: their high heat resistance easily allows straight hair to be curled or curled hair to be straightened using a heating tool such as a curling iron (hereinafter, referred to as "shape imparting properties"), and such a curled or straight shape formed using a heating tool is retained even under hot and humid conditions such as bathing or hair wash (hereinafter, referred to as "shape sustainability"). Furthermore, the wigs using synthetic hair are also excellent because such wigs are more rigid and have higher strength (durability) as compared with wigs using human hair. On the other hand, the wigs using synthetic hair have the following disadvantages: gloss is strong due to smooth surface, and the original purpose of wearing natural feeling of hair with a wig is difficult to achieve due to unnatural texture.

20 **[0004]** Wigs using human hair provide the advantages that the wigs have texture or gloss similar to those of self hair and produce natural appearance upon wearing. Most people who wear wigs do not want to be known to anyone as wearing wigs. Under such circumstances, it is largely advantageous to produce natural appearance on the wig.

[0005] For example, Patent Literature 1 discloses a fiber tress for hair prepared by mixing human hair with polyester fibers having specific physical properties, in order to overcome the disadvantage for poor shape sustainability of human hair while maintaining the excellent characteristics of human hair. Patent Literature 2 discloses a hair ornament product prepared by blending regenerated collagen fibers with human hair, in order to overcome the disadvantage for accumulation of frizz of human hair products and also to supplement permanent wave performance which is a disadvantage of the regenerated collagen fibers.

30 **[0006]**

(Patent Literature 1) International Publication No. WO 2005/037000

(Patent Literature 2) JP-A-2007-177370

Summary of Invention

35 **[0007]** The present invention provides a human hair fiber treatment agent comprised of one or more compositions for treating human hair fibers having free ends at both ends, wherein the human hair fiber treatment agent comprises the following components (A) to (C) in the formulation thereof:

40 (A): at least one formaldehyde derivative selected from the group consisting of formaldehyde, formaldehyde hydrate, glyoxylic acid, glyoxylic acid hydrate, glyoxylate, glyoxal, glyoxal hydrate, glutaraldehyde, and glutaraldehyde hydrate;

(B): phenol compound(s) having electron-donating group(s) on at least one of the meta positions and having hydrogen atom(s) on at least one of the ortho positions and the para position, with the proviso that the electron-donating group at the meta position optionally forms, together with a carbon atom attached thereto, a benzene ring optionally substituted with hydroxy group(s); and

(C): water.

50 **[0008]** The present invention further provides a method for treating human hair fibers, comprising the following step (i):

(i) the step of immersing human hair fibers having free ends at both ends in the human hair fiber treatment agent described above under heating.

55 **[0009]** The present invention further provides a method for producing a wig, comprising the step of treating human hair fibers for wigs by the method for treating human hair fibers described above.

Detailed Description of the Invention

[0010] Human hair currently used in hair ornament products such as wigs undergoes many treatment steps including removal of cuticle by chemical treatment as well as bleaching and dyeing, in order to impart uniform shapes or colors thereto. Therefore, the human hair may be damaged during these steps (Patent Literature 1) and is known to present problems associated with durability (Patent Literature 2).

[0011] In recent years, an increasing number of people have given weight to appearance because of increasing needs for aesthetics. Thus, there has been a demand for wigs having higher durability.

[0012] However, in the techniques described in Patent Literatures 1 and 2, the disadvantages of unnatural appearance and poor feel are still not solved because human hair is mixed with synthetic hair. Thus, these techniques are insufficient for providing natural appearance.

[0013] Thus, the present invention relates to a human hair fiber treatment agent for producing human hair fibers for wigs which are excellent in shape imparting properties, shape sustainability, and durability while retaining the natural appearance of human hair, as well as to a method for treating human hair fibers.

[0014] The present inventors have found that the treatment of human hair fibers for hair implantation to wigs or of a wig having the human hair fibers implanted therewith with a composition containing specific aldehyde compound and specific phenol compound can not only impart shape sustainability to the human hair fibers but also improve the strength of the human hair fibers, thereby improving durability; and that as a result, the treated human hair fibers satisfy shape sustainability, natural gloss, good feel, and high durability simultaneously. Thus the present inventors have completed the invention.

[0015] According to the present invention, human hair fibers for wigs which are excellent in shape sustainability and durability while retaining the natural appearance and good feel of human hair can be produced.

[0016] In the present invention, the human hair fibers having free ends at both ends refer to human hair fibers which are head hair separated from the human head, and are used as an industrial material or a craft material such as hair for wigs, hair extensions, or hair implantation.

[Component (A): specific aldehyde compound]

[0017] The component (A) is at least one formaldehyde derivative selected from the group consisting of formaldehyde, formaldehyde hydrate, glyoxylic acid, glyoxylic acid hydrate, glyoxylate, glyoxal, glyoxal hydrate, glutaraldehyde, and glutaraldehyde hydrate.

[0018] Examples of the formaldehyde hydrate as the component (A) include formaldehyde monohydrate (methanediol). Examples of the glyoxylic acid hydrate include glyoxylic acid monohydrate. Examples of the glyoxal hydrate include glyoxal monohydrate and glyoxal dihydrate. Examples of the glutaraldehyde hydrate include glutaraldehyde monohydrate and glutaraldehyde dihydrate. Examples of salts of these compounds include alkali metal salts such as lithium salt, sodium salt, and potassium salt, and alkaline earth metal salts such as magnesium salt and calcium salt.

[0019] Among these compounds as the component (A), one or more members selected from the group consisting of formaldehyde and glyoxylic acid are preferably contained, and formaldehyde is more preferably contained, in view of imparting higher shape sustainability and durability to the treated human hair fibers.

[0020] The content of the component (A) in the human hair fiber treatment agent of the present invention is preferably 0.1% by mass or more, more preferably 1% by mass or more, further preferably 2.5% by mass or more, still further preferably 5% by mass or more, in view of imparting higher shape sustainability and durability to the treated human hair fibers, and preferably 60% by mass or less, more preferably 50% by mass or less, further preferably 40% by mass or less, still further preferably 35% by mass or less, still further preferably 30% by mass or less, still further preferably 20% by mass or less, still further preferably 15% by mass or less, from the viewpoint mentioned above as well as in view of formulation suitability.

[0021] Specifically, the content of the component (A) in the human hair fiber treatment agent of the present invention is preferably 0.1 to 60% by mass, more preferably 1 to 50% by mass, further preferably 2.5 to 40% by mass, still further preferably 5 to 35% by mass, still further preferably 5 to 30% by mass, still further preferably 5 to 20% by mass, further preferably 5 to 15% by mass, in view of imparting higher shape sustainability and strength to the treated human hair fibers and in view of formulation suitability.

[Component (B): specific phenol compound]

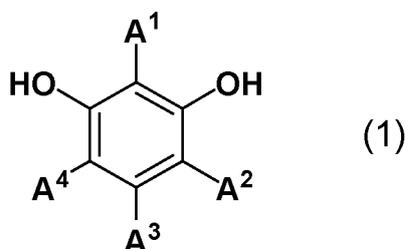
[0022] The component (B) is a phenol compound having electron-donating group(s) on at least one, preferably two, of the meta positions and having hydrogen atom(s) on at least one of the ortho positions and the para position. The electron-donating group at the meta position in the phenol compound optionally forms a benzene ring together with a carbon atom attached thereto, and the benzene ring is optionally further substituted by a hydroxy group. The molecular

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weight of the component (B) is preferably 110 or higher, and preferably 1,000 or lower, more preferably 700 or lower, further preferably 500 or lower, in view of favorable penetration into the human hair fibers. Examples of the phenol compound as the component (B) include the following components (B1), (B2) and (B3):

- 5 (B1) a resorcinol derivative represented by the formula (1);
 (B2) a naphthol derivative represented by the formula (2) or (3); and
 (B3) a flavan-3-ol derivative represented by the formula (4).

10 [0023] The component (B1) is a compound represented by the following formula (1):

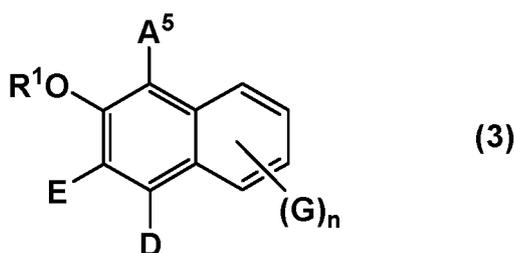
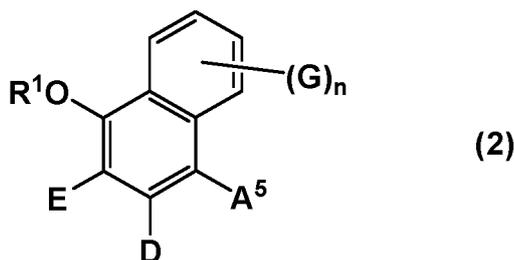


20 wherein

A¹ to A⁴ are the same or different and each represent a hydrogen atom, a hydroxy group, a halogen atom, a carboxyl group or a salt thereof, a sulfonic acid group or a salt thereof, a linear or branched alkyl group or alkenyl group having 1 to 6 carbon atoms, or a linear or branched alkoxy group or alkenyloxy group having 1 to 6 carbon atoms.

25 [0024] Examples of the component (B1) include resorcinol, 2-methylresorcinol, 4-chlororesorcinol, and pyrogallol.

[0025] The component (B2) is a compound represented by the following formula (2) or (3):



wherein

50 R¹ represents a hydrogen atom or a methyl group;

A⁵ represents a hydrogen atom, a linear or branched alkyl group or alkenyl group having 1 to 12 carbon atoms, an optionally substituted aralkyl group or arylalkenyl group having 7 to 12 carbon atoms, a linear or branched alkoxy group or alkenyloxy group having 1 to 6 carbon atoms, a halogen atom or -CO-R² (wherein R² represents a linear or branched alkyl group or alkenyl group having 1 to 12 carbon atoms, an optionally substituted aralkyl group or arylalkenyl group having 7 to 12 carbon atoms or an optionally substituted aromatic hydrocarbon group having 6 to 12 carbon atoms);

D represents a hydrogen atom, a hydroxy group, a methyl group or a linear or branched alkoxy group or alkenyloxy group having 1 to 12 carbon atoms;

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E represents a hydrogen atom, a hydroxy group, a linear or branched alkyl group or alkenyl group having 1 to 6 carbon atoms, or a linear or branched alkoxy group or alkenyloxy group having 1 to 6 carbon atoms;

G represents a hydroxy group, a linear or branched alkyl group or alkenyl group having 1 to 6 carbon atoms, or an alkoxy group having 1 to 6 carbon atoms; and n represents an integer from 0 to 2.

5

[0026] The naphthol derivative represented by the formula (2) or (3) is preferably a compound of the formula (2) or (3) wherein R¹ is a hydrogen atom or an alkyl group or alkenyl having 1 to 4 carbon atoms, more preferably a hydrogen atom.

[0027] A⁵ is preferably a hydrogen atom, a hydroxy group, a linear or branched alkyl group having 1 to 4 carbon atoms or an alkoxy group having 1 to 4 carbon atoms, more preferably a hydrogen atom or a hydroxy group.

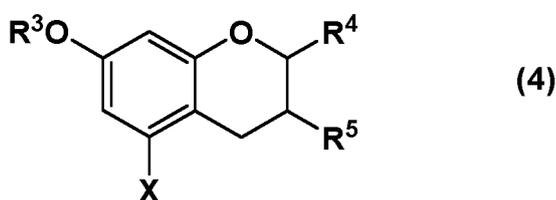
[0028] D is preferably a hydrogen atom, a hydroxy group, a linear or branched alkyl group having 1 to 4 carbon atoms or an alkoxy group having 1 to 4 carbon atoms.

[0029] E is preferably a hydrogen atom, a hydroxy group, an alkyl group having 1 to 4 carbon atoms or an alkoxy group having 1 to 4 carbon atoms.

[0030] Examples of such a compound include 1-naphthol, 2-naphthol, 3-methylnaphthalen-1-ol, naphthalene-1,5-diol, and naphthalene-1,8-diol.

[0031] The compound (B3) is a flavan-3-ol derivative represented by the following formula (4):

20



25

wherein

R³ represents a hydrogen atom or a methyl group;

[0032] X represents a hydrogen atom, a hydroxy group or a methoxy group;

R⁴ represents an aromatic hydrocarbon group optionally substituted by up to three hydroxy groups or methoxy groups and optionally forming a condensed ring with 1,3-dioxolane; and

R⁵ represents a hydroxy group, a methoxy group, an aromatic hydrocarbon group optionally substituted by up to three hydroxy groups or methoxy groups and optionally forming a condensed ring with 1,3-dioxolane, or an arylcarbonyloxy group or an aralkylcarbonyloxy group optionally substituted by up to three hydroxy groups or methoxy groups.

[0032] The molecular weight of the compound represented by the formula (4) is preferably 150 or higher. Also, the molecular weight is preferably 1,000 or lower, further preferably 700 or lower, further preferably 500 or lower, in view of favorable penetration to the inside of the human hair fibers.

[0033] Examples of the component (B3) include catechin, epicatechin, epigallocatechin, catechin gallate, epicatechin gallate, and epigallocatechin gallate. One or more members selected from the group consisting of catechin, epigallocatechin, and epigallocatechin gallate are preferred. A mixture containing any of these compounds, such as green tea extracts, may be used.

[0034] The component (B) is preferably one or two or more members selected from the group consisting of 1-naphthol, 2-naphthol, 3-methylnaphthalen-1-ol, naphthalene-1,5-diol, naphthalene-1,8-diol, catechin, epicatechin, epigallocatechin, catechin gallate, epicatechin gallate, and epigallocatechin gallate, more preferably one or two or more members selected from the group consisting of catechin, epicatechin, epigallocatechin, catechin gallate, epicatechin gallate, and epigallocatechin gallate, in view of more markedly changing the shape of the human hair fibers treated with the human hair fiber treatment agent of the present invention through a condensate of the component (A) and the component (B) formed in the human hair fibers, attaining much better shape sustainability and durability of the human hair fibers, and attaining much better natural appearance and feel of human hair.

[0035] These compounds as the component (B) can be used alone or in combination of two or more thereof, and two or more types of the components (B1) to (B3) may be used in combination. One or more members selected from the group consisting of the compounds as the component (B2) and (B3) are preferred in view of being able to impart higher shape sustainability and durability to the human hair fibers by the configuration of the invention of the present application and in view of enhancing feel.

[0036] The total content of the whole component (B) in the human hair fiber treatment agent of the present invention

is preferably 0.1% by mass or more, more preferably 1% by mass or more, further preferably 2.5% by mass or more, still further preferably 5% by mass or more, in view of imparting higher shape sustainability and durability to the treated human hair fibers, and preferably 60% by mass or less, more preferably 50% by mass or less, further preferably 40% by mass or less, further preferably 35% by mass or less, still further preferably 30% by mass or less, from the viewpoint described above as well as in view of improving hair surface feel.

[0037] Specifically, the content of the component (B) in the human hair fiber treatment agent of the present invention is preferably 0.1 to 60% by mass, more preferably 1 to 50% by mass, further preferably 2.5 to 40% by mass, still further preferably 5 to 35% by mass, still further preferably 5 to 30% by mass, in view of imparting higher shape sustainability and durability to the treated human hair fibers and in view of improving hair surface feel.

[0038] The molar ratio of the component (B) to the component (A), which are applied to the human hair fibers by the human hair fiber treatment agent of the present invention, (B)/(A), is preferably 0.005 or more, more preferably 0.01 or more, further preferably 0.05 or more, still further preferably 0.1 or more, still further preferably 0.5 or more, in view of attaining much better shape sustainability and durability of the treated human hair fibers through a condensate of the component (A) and the component (B) formed in the human hair fibers, and preferably less than 5, more preferably 4 or less, further preferably 3 or less, still further preferably 2 or less, still further preferably 1.8 or less, in view of favorable feel.

[0039] Specifically, the molar ratio of the component (B) to the component (A), (B)/(A), is preferably 0.005 or more and less than 5, more preferably 0.01 to 4, further preferably 0.05 to 3, still further preferably 0.1 to 2, still further preferably 0.5 to 1.8, in view of attaining much better shape sustainability and durability of the treated human hair fibers and in view of favorable feel.

[Component (C): water]

[0040] The human hair fiber treatment agent of the present invention comprises water as a vehicle. The content of the component (C) in the human hair fiber treatment agent of the present invention is preferably 10% by mass or more, more preferably 20% by mass or more, further preferably 30% by mass or more, still further preferably 40% by mass or more, and preferably 99% by mass or less, more preferably 97% by mass or less, further preferably 95% by mass or less, still further preferably 90% by mass or less.

[0041] Specifically, the content of the component (C) in the human hair fiber treatment agent of the present invention is preferably 10 to 99% by mass, more preferably 20 to 97% by mass, further preferably 30 to 95% by mass, still further preferably 40 to 90% by mass.

[0042] The human hair fiber treatment agent of the present invention can be in any of a one-part form and a multiple-part form such as a two-part form. Among them, a multiple-part form, further preferably a two-part form, comprising the component (A) and the component (B) in separate parts is more preferred in view of attaining favorable penetration of the component (A) and the component (B) into the human hair fibers and enhancing the advantageous effects of the present invention. In the case of a multiple-part form, in the present invention, the part containing the component (B) is regarded as the first part, and the part containing the component (A) is regarded as the second part.

[0043] In the case of a multiple-part form, the content of the component (B) in the first part is preferably 0.1% by mass or more, more preferably 1% by mass or more, further preferably 2.5% by mass or more, still further preferably 5% by mass or more, and preferably 50% by mass or less, more preferably 40% by mass or less, further preferably 35% by mass or less, still further preferably 30% by mass or less, in view of imparting higher shape sustainability and durability to the treated human hair fibers.

[0044] Specifically, the content of the component (B) in the first part is preferably 0.1 to 50% by mass, more preferably 1 to 40% by mass, further preferably 2.5 to 35% by mass, still further preferably 5 to 30% by mass, in view of imparting higher shape sustainability and durability to the treated human hair fibers.

[0045] In the case of a multiple-part form, the content of the component (A) in the second part is preferably 0.1% by mass or more, more preferably 1% by mass or more, further preferably 2.5% by mass or more, still further preferably 5% by mass or more, and preferably 50% by mass or less, more preferably 40% by mass or less, further preferably 35% by mass or less, still further preferably 30% by mass or less, in view of imparting higher shape sustainability and durability to the treated human hair fibers.

[0046] Specifically, the content of the component (A) in the second part is preferably 0.1 to 50% by mass, more preferably 1 to 40% by mass, further preferably 2.5 to 35% by mass, still further preferably 5 to 30% by mass, in view of imparting higher shape sustainability and durability to the treated human hair fibers.

[0047] In the case of using the multiple-part form by mixing the first part and the second part, etc., the mixing ratio therebetween is not particularly limited. In the present invention, a composition obtained as a combination by mixing the first part and the second part, etc. preferably serves as the human hair fiber treatment agent of the present invention, in view of attaining favorable penetration of the component (A) and the component (B) into the human hair fibers and enhancing the advantageous effects of the present invention. Thus, in the case of using the multiple-part form by mixing the first part and the second part, etc., the content of each component refers to the content thereof in the composition

as a combination unless otherwise specified.

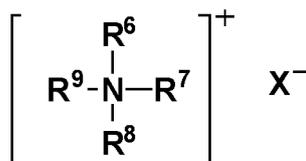
[0048] The human hair fibers may be treated by sequential immersing in the first part and the second part of the multiple-part form. Specifically, this treatment method involves first immersing the human hair fibers in the first part and subsequently immersing the human hair fibers treated with the first part in the second part. In this case, the content can be calculated as a content in a mixed solution supposing that the first part and the second part are mixed in equal amounts. The multiple-part form can contain the component (C) as a solvent in each part, and the component (C) is preferably contained in both the first part and the second part.

[0049] In the case of a one-part form, pH of the part is preferably 5.0 or lower, more preferably 4.8 or lower, further preferably 4.5 or lower, in view of improving penetration into hair, and preferably 0 or higher, more preferably 0.5 or higher, further preferably 1.0 or higher, in view of suppressing hair damage. In the case of a multiple-part form such as a two-part form, pH of each part is preferably 6.0 or lower, more preferably 5.8 or lower, further preferably 5.5 or lower, in view of improving penetration into hair, and preferably 0 or higher, more preferably 0.5 or higher, further preferably 1.0 or higher, in view of suppressing hair damage. The multiple-part form preferably has pH in the range given about the one-part form when the first part and the second part are mixed in equal amounts. The pH according to the present invention is a value determined at 25°C.

[0050] Specifically, in the case of a one-part form, pH of the part is preferably 0 to 5.0, more preferably 0.5 to 4.8, further preferably 1.0 to 4.5, in view of improving penetration into hair and in view of suppressing hair damage. In the case of a multiple-part form such as a two-part form, pH of each part is preferably 0 to 6.0, more preferably 0.5 to 5.8, further preferably 1.0 to 5.5, in view of improving penetration into hair and in view of suppressing hair damage.

[0051] The human hair fiber treatment agent of the present invention preferably contains a cationic surfactant in view of improving the feel of the treated human hair fibers and further improving the advantageous effects of the invention of the present application. The cationic surfactant is preferably a mono long-chain alkyl quaternary ammonium salt having one alkyl group having 8 to 24 carbon atoms and three alkyl groups having 1 to 4 carbon atoms.

[0052] Preferably, at least one mono long-chain alkyl quaternary ammonium surfactant is selected from the group consisting of compounds represented by the following formula:



wherein R⁶ represents a saturated or unsaturated linear or branched alkyl group having 8 to 22 carbon atoms, R¹⁰-CO-NH-(CH₂)_m- or R¹⁰-CO-O-(CH₂)_m- (wherein R¹⁰ represents a saturated or unsaturated linear or branched alkyl chain having 7 to 21 carbon atoms, and m represents an integer of 1 to 4); R⁷, R⁸ and R⁹ each independently represent an alkyl group having 1 to 4 carbon atoms or a hydroxylalkyl group having 1 to 4 carbon atoms; and X⁻ represents a chloride ion, a bromide ion, a methosulfate ion or an ethosulfate ion.

[0053] Preferred examples of the cationic surfactant include long-chain quaternary ammonium compounds such as cetyl trimethyl ammonium chloride, myristyl trimethyl ammonium chloride, behentrimonium chloride, cetyl trimethyl ammonium bromide, and stearamidopropyltrimonium chloride. These cationic surfactants may be used alone or may be used as a mixture.

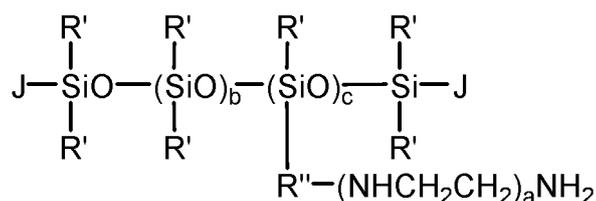
[0054] The content of the cationic surfactant in the human hair fiber treatment agent of the present invention is preferably 0.05% by mass or more, more preferably 0.1% by mass or more, and preferably 10% by mass or less, more preferably 5% by mass or less, in view of improving the feel of the treated human hair fibers and further improving the advantageous effects of the invention of the present application. When the human hair fiber treatment agent is a multiple-part form, the cationic surfactant may be contained in the first part, may be contained in the second part, or may be contained in both the first part and the second part.

[0055] The human hair fiber treatment agent of the present invention preferably contains silicone in view of improving the feel of the treated human hair fibers and improving styling ease. The silicone is preferably one or more members selected from the group consisting of dimethylpolysiloxane and amino-modified silicone.

[0056] Any of cyclic or noncyclic dimethylpolysiloxane polymers can be used as the dimethylpolysiloxane. Examples thereof include SH200 series, BY22-019, BY22-020, BY11-026, B22-029, BY22-034, BY22-050A, BY22-055, BY22-060, BY22-083, and FZ-4188 (all from Dow Corning Toray Co., Ltd.), and KF-9088, KM-900 series, MK-15H, and MK-88 (all from Shin-Etsu Chemical Co., Ltd.).

[0057] Every silicone having an amino group or an ammonium group can be used as the amino-modified silicone. Examples thereof include amino-modified silicone oil with all or some terminal hydroxyl groups end-capped with a methyl group or the like, and amodimethicone without end capping. Preferred examples of the amino-modified silicone include

compounds represented by the following formula, in view of improving the feel of the treated human hair fibers and improving styling ease:



wherein R' represents a hydrogen atom, a hydroxy group or R^X; R^X represents a substituted or unsubstituted monovalent hydrocarbon group having 1 to 20 carbon atoms; J represents R^X, R''-(NHCH₂CH₂)_aNH₂, OR^X or a hydroxy group; R'' represents a divalent hydrocarbon group having 1 to 8 carbon atoms; a represents a number of 0 to 3; and b and c represent numbers, the sum of which becomes 10 or more and less than 20,000, preferably 20 or more and less than 3,000, more preferably 30 or more and less than 1,000, further preferably 40 or more and less than 800, in terms of number average.

[0058] Specific examples of a preferred commercially available product of the amino-modified silicone include: amino-modified silicone oils such as SF8452C and SS3551 (both from Dow Corning Toray Co., Ltd.), and KF-8004, KF-867S, and KF-8015 (all from Shin-Etsu Chemical Co., Ltd.); and amodimethicone emulsions such as SM8704C, SM8904, BY22-079, FZ-4671, and FZ4672 (all from Dow Corning Toray Co., Ltd.).

[0059] The content of the silicone in the human hair fiber treatment agent of the present invention is preferably 0.1% by mass or more, more preferably 0.2% by mass or more, further preferably 0.5% by mass or more, and preferably 20% by mass or less, more preferably 10% by mass or less, further preferably 5% by mass or less, in view of improving the feel of the treated human hair fibers and further improving the advantageous effects of the invention of the present application. When the human hair fiber treatment agent is a multiple-part form, the silicone may be contained in the first part, may be contained in the second part, or may be contained in both the first part and the second part.

[0060] The human hair fiber treatment agent of the present invention preferably contains a cationic polymer in view of improving the feel of the treated human hair fibers.

[0061] The cationic polymer refers to a polymer having a cationic group or a group ionizable with a cationic group and also includes an ampholytic polymer which becomes cationic as a whole. Specific examples thereof include aqueous solutions containing an amino group or an ammonium group at a side chain of the polymer chain, or containing diallyl quaternary ammonium salt as a constituent unit, for example, cationized cellulose derivatives, cationic starch, cationized guar gum derivatives, polymers or copolymers of diallyl quaternary ammonium salt, and quaternized polyvinylpyrrolidone derivatives. Among them, one or two or more members selected from the group consisting of a polymer containing diallyl quaternary ammonium salt as a constituent unit, a quaternized polyvinylpyrrolidone derivative, and a cationized cellulose derivative are preferred, and one or two or more members selected from the group consisting of a polymer or a copolymer of diallyl quaternary ammonium salt and a cationized cellulose derivative are more preferred, in view of improving effects of soft feel upon rinsing or upon shampooing, smoothness and finger combability, styling ease upon drying and moisture-retaining properties, and the stability of the part(s).

[0062] Preferred specific examples of the polymer or copolymer of diallyl quaternary ammonium salt include dimethyl diallyl ammonium chloride polymers (polyquaternium-6, for example, MERQUAT 100; Lubrizol Advanced Materials, Inc.), dimethyl diallyl ammonium chloride/acrylic acid copolymers (polyquaternium-22, for example, MERQUAT 280 and MERQUAT 295; Lubrizol Advanced Materials, Inc.), and dimethyl diallyl ammonium chloride/acrylamide copolymers (polyquaternium-7, for example, MERQUAT 550; Lubrizol Advanced Materials, Inc.).

[0063] Preferred specific examples of the quaternized polyvinylpyrrolidone derivative include polymers obtained by copolymerizing a vinylpyrrolidone copolymer with dimethylaminoethyl methacrylate (polyquaternium 11, for example, GAFQUAT 734, GAFQUAT 755, and GAFQUAT 755N (all from Ashland Inc.)).

[0064] Preferred specific examples of the cationized cellulose include polymers obtained by adding glycidyl trimethyl ammonium chloride to hydroxycellulose (polyquaternium 10, for example, REOGUARD G and REOGUARD GP (all from Lion Corporation.), and Polymer JR-125, Polymer JR-400, Polymer JR-30M, Polymer LR-400, and Polymer LR-30M (all from Amerchol Corp.)), and hydroxyethylcellulose dimethyl diallyl ammonium chloride (polyquaternium-4, for example, CELCOAT H-100 and CELCOAT L-200 (all from Akzo Nobel N.V.)).

[0065] The content of the cationic polymer in the human hair fiber treatment agent of the present invention is preferably 0.001% by mass or more, more preferably 0.01% by mass or more, further preferably 0.05% by mass or more, and preferably 20% by mass or less, more preferably 10% by mass or less, in view of improving the feel of the treated human hair fibers. When the human hair fiber treatment agent is a multiple-part form, the cationic polymer may be contained in the first part, may be contained in the second part, or may be contained in both the first part and the second part.

[0066] The human hair fiber treatment agent of the present invention may further contain an antioxidant at a usual content. The antioxidant can be any antioxidant generally used in the field of hair cosmetics. Examples thereof include ascorbic acid.

5 [Method for treating human hair fibers]

[0067] Human hair fibers can be treated by a method comprising the following step (i) using the human hair fiber treatment agent of the present invention, thereby imparting shape sustainability and high durability to the human hair fibers:

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(i) the step of immersing human hair fibers having free ends at both ends in the human hair fiber treatment agent of the present invention under heating.

[0068] In the step (i), the human hair fibers to be immersed in the human hair fiber treatment agent may be dry or wet. The amount of the human hair fiber treatment agent to immerse the human hair fibers is, in terms of a bath ratio to the mass of the human hair fibers (the mass of the human hair fiber treatment agent / the mass of the human hair fibers), preferably 2 or more, more preferably 3 or more, further preferably 5 or more, further preferably 10 or more, further preferably 20 or more, and preferably 500 or less, more preferably 250 or less, further preferably 100 or less.

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[0069] Specifically, the bath ratio is preferably 2 to 500, more preferably 3 to 250, further preferably 5 to 100, still further preferably 10 to 100, still further preferably 20 to 100.

[0070] In the step (i), the human hair fibers having free ends at both ends may be fixed with a curler or the like in advance and subsequently immersed in the human hair fiber treatment agent of the present invention under heating. This can impart shape sustainability and high durability as well as the desired shape to the human hair fibers.

[0071] The immersing of the human hair fibers in the human hair fiber treatment agent in the step (i) is performed under heating, and this heating is performed by warming the human hair fiber treatment agent. This heating may be performed by immersing the human hair fibers in the human hair fiber treatment agent while heating the human hair fiber treatment agent, or may be performed by immersing the human hair fibers in the human hair fiber treatment agent having a low temperature, followed by heating. The temperature of the human hair fiber treatment agent is preferably 50°C or higher, more preferably 60°C or higher, further preferably 80°C or higher, for increasing the interaction of the component (A) and the component (B) with protein in the human hair fibers, and accelerating condensation reaction between the component (A) and the component (B) in the human hair fibers, thereby obtaining the advantageous effects of the present invention, and preferably lower than 100°C, more preferably 99°C or lower, for preventing operability in subsequent steps from being reduced due to human hair fibers tangled by the vigorous boiling of the treatment agent during heating.

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[0072] The immersing time in the step (i) is appropriately selected depending on the heating temperature used, and is preferably 15 minutes or longer, more preferably 30 minutes or longer, further preferably 1 hour or longer, in view of allowing the human hair fiber treatment agent to penetrate and/or diffuse into the human hair fibers, and causing sufficient polymerization, and preferably 48 hours or shorter, more preferably 24 hours or shorter, further preferably 12 hours or shorter, for suppressing human hair fiber damage.

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[0073] When the human hair fiber treatment agent is a multiple-part form, the first part containing the component (B) and the second part containing the component (A) may be mixed, and then, the human hair fibers can be immersed in this mixture. Alternatively, the human hair fibers may be sequentially immersed in the first part and the second part. In the case of sequential immersing in the first part and the second part, the human hair fibers may be immersed in the first part, kept in this state for a given time, and then immersed in the second part, in view of accelerating the penetration of the human hair fiber treatment agent and enhancing its effects. In this case, the immersing time in the first part is preferably 1 minute or longer, more preferably 3 minutes or longer, further preferably 5 minutes or longer, and preferably 6 hours or shorter, more preferably 4 hours or shorter, further preferably 2 hours or shorter, for allowing the human hair fiber treatment agent to penetrate and/or diffuse into the human hair fibers. In this respect, warming may be performed. This warming is preferably performed at 40 to 90°C. This warming is performed in view of accelerating the penetration of the first part and thus differs from the "heating" in the step (i). When the human hair fiber treatment agent is a multiple-part form, the "heating" in the step (i) refers to heating which is performed after sequential immersing in the first part and the second part or at the same time with immersing in the second part.

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[0074] In the case of sequentially immersing the human hair fibers in the first part and the second part, the step (i) may comprise the step of rinsing off the first part (hereinafter, referred to as an intermediate rinsing step) after immersing in the first part and before immersing in the second part. Preferably, the step (i) does not comprise the intermediate rinsing step in view of shortening a treatment time.

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[0075] The step (i) is preferably performed in an environment where the evaporation of moisture is suppressed, in view of imparting higher shape sustainability and durability to the treated human hair fibers. Specific examples of the

approach of suppressing the evaporation of moisture include a method of covering a container for the human hair fiber treatment agent with the human hair fibers immersed therein with a film-like substance, a cap, a lid, or the like made of a material impermeable to water vapor.

[0076] After the step (i), the human hair fibers may or may not be rinsed, and are preferably rinsed in view of preventing the feel of the human hair fibers from being reduced due to redundant polymerization products.

[0077] These treatments allow the components (A) and (B) to penetrate into the human hair fibers, presumably causing their interaction with protein in the human hair fibers. Also, a condensate of the component (A) and the component (B) is formed in the human hair fibers. Hence, the human hair fibers treated by the method of the present invention are less likely to lose shape even when cleansed.

[0078] After the step (i), the following step (ii) may be further performed and can thereby markedly improve hair surface feel:

(ii) the step of immersing the human hair fibers in a surface finish agent (I) comprising components (B1) and (C).

[0079] In this case, the preferred component (B1) is the same as that for the human hair fiber treatment agent mentioned above. The content of the component (B1) in the surface finish agent (I) is preferably 1% by mass or more, more preferably 2.5% by mass or more, further preferably 5% by mass or more, still further preferably 10% by mass or more, still further preferably 20% by mass or more, and preferably 95% by mass or less, more preferably 90% by mass or less, further preferably 88% by mass or less, still further preferably 85% by mass or less, still further preferably 80% by mass or less.

[0080] Specifically, the content of the component (B1) in the surface finish agent (I) is preferably 1 to 95% by mass, more preferably 2.5 to 90% by mass, further preferably 5 to 88% by mass, still further preferably 10 to 85% by mass, still further preferably 20 to 80% by mass.

[0081] The surface finish agent (I) may contain an additional component without impairing the advantageous effects of the present invention. The content of the additional component is preferably 3% by mass or less, more preferably 1% by mass or less, further preferably 0.1% by mass or less, still further preferably substantially 0% by mass, in view of improving hair surface feel.

[0082] pH of the surface finish agent (I) is preferably 7.0 or lower, more preferably 6.8 or lower, further preferably 6.5 or lower, in view of improving penetration into hair, and preferably 0 or higher, more preferably 0.5 or higher, further preferably 1.0 or higher, in view of suppressing hair damage.

[0083] Specifically, pH of the surface finish agent (I) is preferably 0 to 7.0, more preferably 0.5 to 6.8, further preferably 1.0 to 6.5, in view of improving penetration into hair and in view of suppressing hair damage.

[0084] The immersing time of the human hair fibers in the surface finish agent (I) in the step (ii) is preferably 1 hour or longer, more preferably 3 hours or longer, further preferably 6 hours or longer, still further preferably 24 hours or longer, and preferably 1 month or shorter, more preferably 2 weeks or shorter, further preferably 10 days or shorter, still further preferably 168 hours or shorter, for allowing the surface finish agent (I) to penetrate and/or diffuse into the human hair fibers. In this respect, warming may be performed. The warming temperature is preferably 20 to 90°C.

[0085] In the step (ii), the human hair fibers to be immersed in the surface finish agent (I) may be dry or wet. The amount of the surface finish agent (I) to immerse the human hair fibers is, in terms of a bath ratio to the mass of the human hair fibers (the mass of the surface finish agent (I) / the mass of the human hair fibers treated in the step (i)), preferably 2 or more, more preferably 3 or more, further preferably 5 or more, further preferably 10 or more, further preferably 20 or more, and preferably 500 or less, more preferably 250 or less, further preferably 100 or less.

[0086] Specifically, the bath ratio is preferably 2 to 500, more preferably 3 to 250, further preferably 5 to 100, further preferably 10 to 100, still further preferably 20 to 100.

[Optional treatment to be further added]

[0087] The method for treating human hair fibers according to the present invention may additionally comprise one or more treatments selected from the group consisting of cuticle removal, bleaching, hair dyeing, and surface finish for hydrophobization and/or reduction in friction, in addition to the steps (i) and (ii) mentioned above.

[0088] In this respect, each treatment of cuticle removal, bleaching, and hair dyeing may be performed before or after the steps (i) and (ii) mentioned above or may be performed between the steps among the steps (i) to (iii). Also, a plurality of treatments may be added in combination. Any of the treatments may be performed first, except that the bleaching needs to be performed before the hair dyeing in the case of adding both bleaching and hair dyeing. A different treatment such as cuticle removal may be performed between the bleaching and the hair dyeing.

[0089] On the other hand, the surface finish for hydrophobization and/or reduction in friction needs to be performed after the step (i) mentioned above, or to be performed after the step (ii) when performed in combination with surface finish for improvement in feel in the step (ii). Among others, the surface finish for hydrophobization and/or reduction in friction additionally performed after the step (ii) can produce more favorable results. The surface finish for hydrophobization and/or reduction in friction is not particularly limited by the order of treatments also including cuticle removal, bleaching, and hair dyeing as long as the surface finish is performed at a stage after the step (i), (ii) or (iii) as described

above.

(Cuticle removal)

5 **[0090]** Cuticle present on human hair fiber surface has a directional structure where scales are piled. If human hair fibers having different directions coexist in one fiber tress, these human hair fibers get stuck with each other and are easily tangled, markedly impairing workability. Hence, the cuticle removal is performed in order to remove hair direction for easy handling, and performed by immersing the human hair fibers in a composition for cuticle removal given below.

10 **[0091]** The composition for cuticle removal contains a chlorine precursor having a cuticle peeling effect, and water. Examples of the chlorine precursor include sodium hypochlorite, potassium dichloroisocyanurate, sodium dichloroisocyanurate, and trichloroisocyanuric acid. One or more chlorine precursors selected from the group consisting of these compounds can be used.

15 **[0092]** The content of the chlorine precursor in the composition for cuticle removal is preferably 1% by mass or more, more preferably 2% by mass or more, and preferably 15% by mass or less, more preferably 12% by mass or less, further preferably 9% by mass or less.

15 **[0093]** pH at 25°C of the composition for cuticle removal is preferably 2 or higher, more preferably 3 or higher, further preferably 4.5 or higher, and preferably 10 or lower, more preferably 8 or lower, further preferably 7 or lower.

(Bleaching)

20 **[0094]** The bleaching is performed by immersing the human hair fibers in a bleach composition containing an alkali agent, an oxidizing agent and water. The bleach composition is usually of two-part type. The first part contains the alkali agent and water, and the second part contains the oxidizing agent and water. These two parts are usually stored separately and mixed before immersing of hair.

25 **[0095]** Preferred examples of the alkali agent include, but are not limited to: ammonia and salts thereof; alkanolamines (monoethanolamine, isopropanolamine, 2-amino-2-methylpropanol, 2-aminobutanol, etc.) and salts thereof; alkanediamines (1,3-propanediamine, etc.) and salts thereof; and carbonates (guanidine carbonate, sodium carbonate, potassium carbonate, sodium bicarbonate, potassium bicarbonate, etc.); and mixtures thereof.

30 **[0096]** The content of the alkali agent in the bleach composition (in the case of two-part type, in a mixture of the first part and the second part) is preferably 0.1% by mass or more, more preferably 0.5% by mass or more, further preferably 1% by mass or more, and preferably 15% by mass or less, more preferably 10% by mass or less, further preferably 7.5% by mass or less.

35 **[0097]** Preferred examples of the oxidizing agent include, but are not limited to, hydrogen peroxide, urea peroxide, melamine peroxide and sodium bromate. Among these oxidizing agents, hydrogen peroxide is preferred.

35 **[0098]** The content of the oxidizing agent in the bleach composition is preferably 1% by mass or more, more preferably 2% by mass or more, and preferably 15% by mass or less, more preferably 12% by mass or less, further preferably 9% by mass or less.

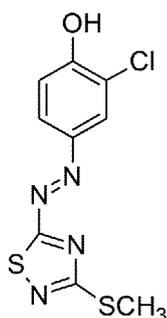
40 **[0099]** In the case of separately storing the first part and the second part, pH at 25°C of the second part is preferably 2 or higher, more preferably 2.5 or higher, and preferably 6 or lower, more preferably 4 or lower. This pH can be adjusted with a suitable buffer. pH at 25°C of the bleach composition is preferably 6 or higher, more preferably 6.5 or higher, further preferably 6.8 or higher, and preferably 11 or lower, more preferably 10.5 or lower, further preferably 10 or lower.

(Hair dyeing)

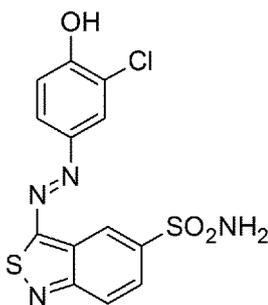
45 **[0100]** The hair dyeing is performed by immersing the human hair fibers in a hair dye composition. The hair dye composition contains a dye and can optionally contain an alkali agent or an acid, an oxidizing agent, or the like. Examples of the dye include direct dyes, oxidizing dyes and combinations thereof.

50 **[0101]** The type of the direct dye is not particularly limited, and an arbitrary direct dye suitable for hair dyeing can be used. Examples of the direct dye include anionic dyes, nitro dyes, disperse dyes, cationic dyes, and dyes having an azophenol structure, selected from the group consisting of the following HC Red 18, HC Blue 18 and HC Yellow 16, and salts thereof, and mixtures thereof.

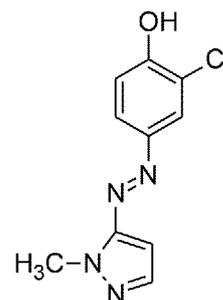
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HC Red 18



HC Blue 18



HC Yellow 16

15 **[0102]** Examples of the cationic dye include, but are not limited to, Basic Blue 6, Basic Blue 7, Basic Blue 9, Basic Blue 26, Basic Blue 41, Basic Blue 99, Basic Brown 4, Basic Brown 16, Basic Brown 17, Natural Brown 7, Basic Green 1, Basic Orange 31, Basic Red 2, Basic Red 12, Basic Red 22, Basic Red 51, Basic Red 76, Basic Violet 1, Basic Violet 2, Basic Violet 3, Basic Violet 10, Basic Violet 14, Basic Yellow 57 and Basic Yellow 87 and mixtures thereof. Basic Red 51, Basic Orange 31, Basic Yellow 87 and mixtures thereof are particularly preferred.

20 **[0103]** Examples of the anionic dye include, but are not limited to, Acid Black 1, Acid Blue 1, Acid Blue 3, Food Blue 5, Acid Blue 7, Acid Blue 9, Acid Blue 74, Acid Orange 3, Acid Orange 4, Acid Orange 6, Acid Orange 7, Acid Orange 10, Acid Red 1, Acid Red 14, Acid Red 18, Acid Red 27, Acid Red 33, Acid Red 50, Acid Red 52, Acid Red 73, Acid Red 87, Acid Red 88, Acid Red 92, Acid Red 155, Acid Red 180, Acid Violet 2, Acid Violet 9, Acid Violet 43, Acid Violet 49, Acid Yellow 1, Acid Yellow 10, Acid Yellow 23, Acid Yellow 3, Food Yellow No. 8, D&C Brown No. 1, D&C Green No. 5, D&C Green No. 8, D&C Orange No. 4, D&C Orange No. 10, D&C Orange No. 11, D&C Red No. 21, D&C Red No. 27, D&C Red No. 33, D&C Violet 2, D&C Yellow No. 7, D&C Yellow No. 8, D&C Yellow No. 10, FD&C Red 2, FD&C Red 40, FD&C Red No. 4, FD&C Yellow No. 6, FD&C Blue 1, Food Black 1, Food Black 2, and alkali metal salts (sodium salt, potassium salt, etc.) thereof and mixtures thereof.

30 **[0104]** Among them, the anionic dye is preferably selected from the group consisting of Acid Black 1, Acid Red 52, Acid Violet 2, Acid Violet 43, Acid Red 33, Acid Orange 4, Acid Orange 7, Acid Red 27, Acid Yellow 3 and Acid Yellow 10, and salts thereof. The anionic dye is more preferably selected from the group consisting of Acid Red 52, Acid Violet 2, Acid Red 33, Acid Orange 4 and Acid Yellow 10, and salts thereof and mixtures thereof.

35 **[0105]** Examples of the nitro dye include, but are not limited to, HC Blue No. 2, HC Blue No. 4, HC Blue No. 5, HC Blue No. 6, HC Blue No. 7, HC Blue No. 8, HC Blue No. 9, HC Blue No. 10, HC Blue No. 11, HC Blue No. 12, HC Blue No. 13, HC Brown No. 1, HC Brown No. 2, HC Green No. 1, HC Orange No. 1, HC Orange No. 2, HC Orange No. 3, HC Orange No. 5, HC Red BN, HC Red No. 1, HC Red No. 3, HC Red No. 7, HC Red No. 8, HC Red No. 9, HC Red No. 10, HC Red No. 11, HC Red No. 13, HC Red No. 54, HC Red No. 14, HC Violet BS, HC Violet No. 1, HC Violet No. 2, HC Yellow No. 2, HC Yellow No. 4, HC Yellow No. 5, HC Yellow No. 6, HC Yellow No. 7, HC Yellow No. 8, HC Yellow No. 9, HC Yellow No. 10, HC Yellow No. 11, HC Yellow No. 12, HC Yellow No. 13, HC Yellow No. 14, HC Yellow No. 15, 2-amino-6-chloro-4-nitrophenol, picramic acid, 1,2-diamino-4-nitrobenzole, 1,4-diamino-2-nitrobenzole, 3-nitro-4-aminophenol, 1-hydroxy-2-amino-3-nitrobenzole and 2-hydroxyethylpicramic acid and mixtures thereof.

40 **[0106]** Examples of the disperse dye include, but are not limited to, Disperse Blue 1, Disperse Black 9 and Disperse Violet 1 and mixtures thereof.

45 **[0107]** These direct dyes can be used alone or in combination of two or more thereof, and direct dyes differing in ionicity may be used in combination.

[0108] The content of the direct dye in the hair dye composition is preferably 0.001% by mass or more, more preferably 0.01% by mass or more, further preferably 0.05% by mass or more, in view of obtaining sufficient hair dyeing properties, and preferably 10% by mass or less, more preferably 7.5% by mass or less, further preferably 5.0% by mass or less, further preferably 3.0% by mass or less, in view of blendability.

50 **[0109]** When the hair dye composition contains only a direct dye as the dye, no oxidizing agent is necessary for dyeing hair. In the case of lightening the color of hair, the composition may contain an oxidizing agent.

[0110] When the hair dye composition contains an oxidizing dye, the hair dye composition is usually of two-part type. The first part contains an oxidizing dye intermediate (precursor and coupler) and an alkali agent, and the second part contains an oxidizing agent such as hydrogen peroxide. These two parts are usually stored separately and mixed before immersing of hair.

55 **[0111]** The oxidizing dye intermediate is not particularly limited, and any known precursor and coupler usually used in hair dyeing products can be suitably used.

[0112] Examples of the precursor include, but are not limited to, paraphenylenediamine, toluene-2,5-diamine, 2-chloro-

paraphenylenediamine, N-methoxyethyl-paraphenylenediamine, N-phenylparaphenylenediamine, N,N-bis(2-hydroxyethyl)-paraphenylenediamine, 2-(2-hydroxyethyl)-paraphenylenediamine, 2,6-dimethyl-paraphenylenediamine, 4,4'-diaminodiphenylamine, 1,3-bis(N-(2-hydroxyethyl)-N-(4-aminophenyl)amino)-2-propanol, PEG-3,3,2'-paraphenylenediamine, paraaminophenol, paramethylaminophenol, 3-methyl-4-aminophenol, 2-aminomethyl-4-aminophenol, 2-(2-hydroxyethylaminomethyl)-4-aminophenol, orthoaminophenol, 2-amino-5-methylphenol, 2-amino-6-methylphenol, 2-amino-5-acetamidophenol, 3,4-diaminobenzoic acid, 5-aminosalicylic acid, 2,4,5,6-tetraaminopyrimidine, 2,5,6-triamino-4-hydroxypyrimidine, 4,5-diamino-1-(4'-chlorobenzyl)pyrazole, 4,5-diamino-1-hydroxyethylpyrazole, and salts of these substances and mixtures thereof.

[0113] Examples of the coupler include, but are not limited to, metaphenylenediamine, 2,4-diaminophenoxyethanol, 2-amino-4-(2-hydroxyethylamino)anisole, 2,4-diamino-5-methylphenetole, 2,4-diamino-5-(2-hydroxyethoxy)toluene, 2,4-dimethoxy-1,3-diaminobenzene, 2,6-bis(2-hydroxyethylamino)toluene, 2,4-diamino-5-fluorotoluene, 1,3-bis(2,4-diaminophenoxy)propane, metaaminophenol, 2-methyl-5-aminophenol, 2-methyl-5-(2-hydroxyethylamino)phenol, 2,4-dichloro-3-aminophenol, 2-chloro-3-amino-6-methylphenol, 2-methyl-4-chloro-5-aminophenol, N-cyclopentyl-metaaminophenol, 2-methyl-4-methoxy-5-(2-hydroxyethylamino)phenol, 2-methyl-4-fluoro-5-aminophenol, paraaminoorthocresol, resorcinol, 2-methylresorcinol, 4-chlororesorcinol, 1-naphthol, 1,5-dihydroxynaphthalene, 1,7-dihydroxynaphthalene, 2,7-dihydroxynaphthalene, 2-isopropyl-5-methylphenol, 4-hydroxyindole, 5-hydroxyindole, 6-hydroxyindole, 7-hydroxyindole, 6-hydroxybenzomorpholine, 3,4-methylenedioxyphenol, 2-bromo-4,5-methylenedioxyphenol, 3,4-methylenedioxyaniline, 1-(2-hydroxyethyl)amino-3,4-methylenedioxybenzene, 2,6-dihydroxy-3,4-dimethylpyridine, 2,6-dimethoxy-3,5-diaminopyridine, 2,3-diamino-6-methoxypyridine, 2-methylamino-3-amino-6-methoxypyridine, 2-amino-3-hydroxypyridine, 2,6-diaminopyridine, and salts of these substances and mixtures thereof.

[0114] The content of each of the precursor and the coupler in the hair dye composition is preferably 0.01% by mass or more, more preferably 0.1% by mass or more, and preferably 10% by mass or less, more preferably 7.5% by mass or less, further preferably 5% by mass or less.

[0115] The hair dye composition containing an oxidizing dye further contains an alkali agent. Preferred examples of the alkali agent include, but are not limited to: ammonia and salts thereof; alkanolamines (monoethanolamine, isopropanolamine, 2-amino-2-methylpropanol, 2-aminobutanol, etc.) and salts thereof; alkanediamines (1,3-propanediamine, etc.) and salts thereof; and carbonates (guanidine carbonate, sodium carbonate, potassium carbonate, sodium bicarbonate, potassium bicarbonate, etc.); and mixtures thereof.

[0116] The content of the alkali agent in the hair dye composition is preferably 0.1% by mass or more, more preferably 0.5% by mass or more, further preferably 1% by mass or more, and preferably 15% by mass or less, more preferably 10% by mass or less, further preferably 7.5% by mass or less.

[0117] When the hair dye composition contains an oxidizing dye, the composition (second part) containing the oxidizing agent is stored separately from the composition (first part) containing the oxidizing dye, and mixed therewith before immersing of hair. Preferred examples of the oxidizing agent include, but are not limited to, hydrogen peroxide, urea peroxide, melamine peroxide and sodium bromate. Among these oxidizing agents, hydrogen peroxide is preferred.

[0118] The content of the oxidizing agent in the hair dye composition is preferably 1% by mass or more, more preferably 2% by mass or more, and preferably 15% by mass or less, more preferably 12% by mass or less, further preferably 9% by mass or less.

[0119] In the case of separately storing the first part and the second part, pH at 25°C of the second part is preferably 2 or higher, more preferably 2.5 or higher, and preferably 6 or lower, more preferably 4 or lower. This pH can be adjusted with a suitable buffer. pH at 25°C of the hair dye composition prepared by mixing the first part and the second part is preferably 6 or higher, more preferably 6.5 or higher, further preferably 6.8 or higher, and preferably 11 or lower, more preferably 10.5 or lower, further preferably 10 or lower.

[0120] The hair dye composition containing an oxidizing dye may further contain the direct dye listed above.

[0121] The hair dye composition can preferably further contain a surfactant, a conditioning component, or the like given below, and can preferably be in any of solution, emulsion, cream, paste and mousse forms.

[0122] The temperature of the hair dye composition is preferably 0°C or higher, more preferably 10°C or higher, further preferably 20°C or higher, and preferably 90°C or lower, more preferably 80°C or lower, in view of allowing the hair dye composition to efficiently penetrate and/or diffuse to the inside of the human hair fibers, and further enhancing the effects of hair dyeing.

(Surface finish for hydrophobization and/or reduction in friction)

[0123] The surface finish for hydrophobization and/or reduction in friction is performed by immersing hair in a surface finish agent (II) given below at a stage after the step (i) mentioned above, or at a stage after the step (ii) when surface finish for improvement in feel in the step (ii) is performed.

[0124] The surface finish agent (II) contains 0.01% by mass or more and 5.00% by mass or less of the following component (D), and water:

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(D) an epoxyaminosilane copolymer which is a reaction product of the following compounds (a) to (d):

- (a) polysiloxane having at least two oxiranyl groups or oxetanyl groups;
 (b) polyether having at least two oxiranyl groups or oxetanyl groups;
 (c) aminopropyltrialkoxysilane; and
 (d) a compound selected from the group consisting of the following primary and secondary amines:

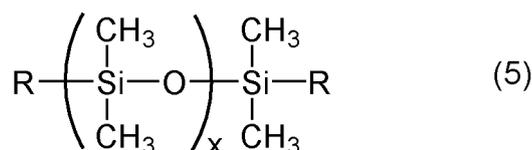
primary amine: methylamine, ethylamine, propyleneamine, ethanolamine, isopropylamine, butylamine, isobutylamine, hexylamine, dodecylamine, oleylamine, aniline, aminopropyltrimethylsilane, aminopropyltriethylsilane, aminomorpholine, aminopropyldiethylamine, benzylamine, naphthylamine, 3-amino-9-ethylcarbazole, 1-aminoheptafluorohexane, and 2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-pentadecafluoro-1-octanamine; and
 secondary amine: methylethylamine, methyloctadecylamine, diethanolamine, dibenzylamine, dihexylamine dicyclohexylamine, piperidine, pyrrolidine phthalimide, and polymer amine.

[Component (D): epoxyaminosilane copolymer]

[0125] The epoxyaminosilane copolymer as the component (D) is a reaction product of the following compounds (a) to (d).

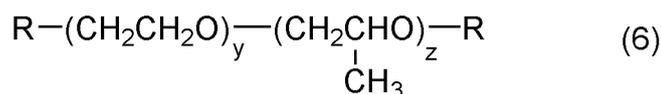
<Compounds (a) and (b)>

[0126] The compound (a) is polysiloxane containing at least two oxiranyl groups or oxetanyl groups. Examples thereof include compounds represented by the following formula (5):



wherein R represents a hydrocarbon group having 1 to 6 carbon atoms and terminally having an oxiranyl group or an oxetanyl group, and optionally containing a heteroatom; and x represents a number of 1 to 1,000.

[0127] The compound (b) is polyether containing at least two oxiranyl groups or oxetanyl groups. Examples thereof include compounds represented by the following formula (6):



wherein R is as defined above; y represents a number of 1 to 100; z represents a number of 0 to 100; and y + z is 1 to 200.

[0128] In the formulas (5) and (6), the heteroatom optionally contained in R is preferably an oxygen atom. Examples of R include an oxiranylmethyl group (glycidyl group), an oxiranylmethoxy group (glycidyoxy group), an oxiranylmethoxypropyl group (glycidyoxypropyl group), an oxetanylmethyl group, an oxetanylmethoxy group, an oxetanylmethoxypropyl group, and a 3-ethyloxetanylmethyl group. Among them, a hydrocarbon group having 1 to 4 carbon atoms and having an oxiranyl group, and optionally containing a hetero oxygen atom is preferred, and at least one member selected from the group consisting of an oxiranylmethyl group (glycidyl group), an oxiranylmethoxy group (glycidyoxy group), and an oxiranylmethoxypropyl group (glycidyoxypropyl group) is further preferred.

<Compound (c)>

[0129] The compound (c) is aminopropyltrialkoxysilane. Examples of the alkoxy group in the compound (c) include alkoxy groups having 1 to 6 carbon atoms. An alkoxy group having 2 to 4 carbon atoms, further preferably an alkoxy group having 3 carbon atoms, is preferred. Among them, an isopropoxy group is preferred. Examples of the compound (c) include aminopropyltrimethoxysilane, aminopropyltriethoxysilane, aminopropyltripropoxysilane, aminopropyltriisopropoxysilane, aminopropyltributoxysilane, and aminopropyl tri-tert-butoxysilane. Among them, aminopropyltriisopropoxysilane is preferred. Any of these compounds (c) can be used alone or in combination of two or more thereof.

<Compound (d)>

[0130] The compound (d) is a compound selected from the group consisting of the following primary and secondary amines:

primary amine: methylamine, ethylamine, propyleneamine, ethanolamine, isopropylamine, butylamine, isobutylamine, hexylamine, dodecylamine, oleylamine, aniline, aminopropyltrimethylsilane, aminopropyltriethylsilane, aminomorpholine, aminoethyldimethylamine, aminoethyldiethylamine, aminoethyldibutylamine, aminopropylidimethylamine, aminopropylidethylamine, aminopropylidibutylamine, benzylamine, naphthylamine, 3-amino-9-ethylcarbazole, 1-aminoheptafluorohexane, and 2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-pentadecafluoro-1-octanamine; and secondary amine: methylethylamine, methyloctadecylamine, diethanolamine, dibenzylamine, dihexylamine dicyclohexylamine, piperidine, pyrrolidine phthalimide, and polymer amine.

[0131] Among them, primary amine is preferred, and one member selected from the group consisting of aminopropylidethylamine, aminopropylidimethylamine, and aminopropylidibutylamine is further preferred. Any of these compounds (d) can be used alone or in combination of two or more thereof.

[0132] The reaction of the compounds (a) to (d) is performed, for example, by refluxing these compounds for a given time in a solvent such as isopropanol. In this context, the molar ratio of the oxiranyl groups or the oxetanyl groups of the compounds (a) and (b) to the amino group of the compound (c) is preferably 1 or more, more preferably 1.1 or more, further preferably 1.2 or more, and preferably 4 or less, more preferably 3.9 or less, further preferably 3.8 or less.

[0133] Examples of the component (D) include compounds under INCI name of polysilicone-29. Examples of a commercially available product thereof include Silsoft CLX-E from Momentive Performance Materials, Inc. (active component: 15% by mass, containing dipropylene glycol and water).

[0134] The content of the component (D) in the surface finish agent (II) is 0.01% by mass or more, preferably 0.05% by mass or more, more preferably 0.10% by mass or more, further preferably 0.20% by mass or more, in view of imparting sufficient hydrophobicity to the human hair fibers, and 5.00% by mass or less, preferably 4.00% by mass or less, more preferably 3.00% by mass or less, further preferably 2.00% by mass or less, in view of offering no sticky feel.

[pH adjuster]

[0135] pH at 25°C of the surface finish agent (II) is preferably in a range given below in view of enhancing the reaction rate of the trialkoxysilane moiety of the component (D) in an acidic region or a basic region. When the surface finish agent (II) has a pH in an acidic region, pH is preferably 1 or higher, more preferably 1.5 or higher, further preferably 2 or higher, and preferably 5 or lower, more preferably 4.0 or lower, further preferably 3.5 or lower. When the surface finish agent (II) has a pH in a basic region, pH is preferably 7 or higher, more preferably 7.5 or higher, further preferably 8.0 or higher, and preferably 11 or lower, more preferably 10.5 or lower, further preferably 10 or lower. In order to adjust pH of the surface finish agent (II) to the range described above, the surface finish agent (II) can appropriately contain a pH adjuster. Examples of the pH adjuster that can be used include alkali agents including: alkanolamines such as monoethanolamine, isopropanolamine, 2-amino-2-methylpropanol, and 2-aminobutanol, or salts thereof; alkanediamines such as 1,3-propanediamine, or salts thereof; carbonates such as guanidine carbonate, sodium carbonate, potassium carbonate, sodium bicarbonate, and potassium bicarbonate; and hydroxides such as sodium hydroxide and potassium hydroxide. Examples of the pH adjuster that can be used include acid agents including: inorganic acids such as hydrochloric acid and phosphoric acid; hydrochlorides such as monoethanolamine hydrochloride; phosphates such as monopotassium dihydrogen phosphate, and disodium monohydrate phosphate; and organic acids such as lactic acid and malic acid.

[0136] The amount of the surface finish agent (II) to immerse the human hair fibers is, in terms of a bath ratio to the mass of the human hair fibers (the mass of the surface finish agent (II) / the mass of the human hair fibers), preferably 2 or more, more preferably 5 or more, further preferably 10 or more, and preferably 100 or less, more preferably 50 or less, further preferably 20 or less.

[0137] The treatment of the human hair fibers by the method for treating human hair fibers described above can produce human hair fibers for wigs which are excellent in shape sustainability and durability while retaining the natural appearance of human hair. Also, a wig can be produced using the fibers.

[0138] The treatment of the human hair fibers by the method for treating human hair fibers according to the present invention can produce human hair fibers for hair extensions which are excellent in shape sustainability and durability while retaining the natural appearance of human hair. Also, hair extensions can be produced using the fibers.

[0139] The following aspects are preferred for the treatment agent capable of producing human hair fibers for wigs which are excellent in shape sustainability and durability while retaining the natural appearance and good feel of human hair, and a treatment method using the treatment agent.

[Treatment agent 1]

5 **[0140]** A human hair fiber treatment agent comprised of one or more compositions for treating human hair fibers having free ends at both ends, wherein the human hair fiber treatment agent comprises the following components (A) to (C) and ethanol in the formulation thereof and has pH of 1.0 to 4.5:

- 10 (A) 5 to 15% by mass of formaldehyde or glyoxal;
(B) 10 to 20% by mass of resorcinol or 1-naphthol;
(C) a balance of water; and

0 to 40% by mass of ethanol.

[Treatment method 1]

15 **[0141]** A method for treating human hair fibers, comprising the following step (i):

(i) the step of immersing human hair fibers having free ends at both ends in a human hair fiber treatment agent under heating of 90 to 99°C for 1 to 3 hours at a bath ratio of (human hair fiber treatment agent mass) / (human hair fiber mass) = 20 to 100, wherein

20 the human hair fiber treatment agent comprised of one or more compositions, wherein the human hair fiber treatment agent comprises the following components (A) to (C) and ethanol in the formulation thereof and has pH of 1.0 to 4.5:

- 25 (A) 5 to 15% by mass of formaldehyde or glyoxal;
(B) 10 to 20% by mass of resorcinol or 1-naphthol;
(C) a balance of water; and

0 to 40% by mass of ethanol.

[Treatment method 1']

30 **[0142]** The method for treating human hair fibers, wherein the treatment method 1 further comprises the following step (ii) after the step (i):

35 (ii) the step of immersing the human hair fibers treated in the step (i) in a surface finish agent (I) at a temperature of 20 to 90°C for 24 to 168 hours at a bath ratio of (the mass of the surface finish agent (I)) / (the mass of the human hair fibers treated in the step (i)) = 20 to 100, wherein the surface finish agent (I) comprises the following components (B1) and (C) and has pH of 1.0 to 6.5:

- 40 (B1) 20 to 80% by mass of resorcinol; and
(C) a balance of water.

[Treatment agent 2]

45 **[0143]** A human hair fiber treatment agent for treating human hair fibers having free ends at both ends, the human hair fiber treatment agent being in a multiple-part form comprising the following first part and second part, wherein the human hair fiber treatment agent has pH of 2.0 to 5.0 when the first part and the second part are mixed in equal amounts:

the first part comprising the following components (B) and (C) and having pH of 2.0 to 5.5:

- 50 (B) 5 to 30% by mass of any one member selected from the group consisting of catechin, epigallocatechin and epigallocatechin gallate; and
(C) a balance of water; and

the second part comprising the following components (A) and (C) and ethanol and having pH of 1.0 to 4.5:

- 55 (A) 5 to 15% by mass of formaldehyde or glyoxal;
(C) a balance of water; and
0 to 40% by mass of ethanol.

[Treatment agent 2']

5 **[0144]** A human hair fiber treatment agent kit comprising a first part and a second part of the treatment agent 2 and further comprising a surface treatment agent (I) comprising 20 to 80% by mass of resorcinol and water and having pH of 1 to 6.5.

[Treatment method 2]

10 **[0145]** A method for treating human hair fibers, comprising the following steps (i-1) and (i-2):

(i-1) the step of immersing human hair fibers having free ends at both ends in a first part under heating of 90 to 99°C for 1 to 3 hours at a bath ratio of (human hair fiber treatment agent mass) / (human hair fiber mass) = 20 to 100, wherein the first part comprises the following components (B) and (C) and has pH of 2.0 to 5.5:

15 (B) 5 to 30% by mass of any one member selected from the group consisting of catechin, epigallocatechin, and epigallocatechin gallate; and
(C) a balance of water; and

20 (i-2) the step of immersing the human hair fibers treated in the step (i-1) in a second part under heating of 90 to 99°C for 1 to 3 hours at a bath ratio of (second part mass) / (the mass of the human hair fibers treated in the step (i-1)) = 20 to 100, wherein the second part comprises the following components (A) and (C) and ethanol and has pH of 1.0 to 4.5:

25 (A) 5 to 15% by mass of formaldehyde or glyoxal;
(C) a balance of water; and
0 to 40% by mass of ethanol.

[Treatment method 2']

30 **[0146]** The method for treating human hair fibers, wherein the treatment method 2 further comprises the following step (ii) after the step (i):

35 (ii) the step of immersing the human hair fibers treated in the step (i) in a surface finish agent (I) at a temperature of 20 to 90°C for 24 to 168 hours at a bath ratio of (the mass of the surface finish agent (I)) / (the mass of the human hair fibers treated in the step (i)) = 20 to 100, wherein the surface finish agent (I) comprises the following components (B1) and (C) and has pH of 1.0 to 6.5:

40 (B1) 20 to 60% by mass of resorcinol; and
(C) a balance of water.

[0147] Hereinafter, preferred aspects of the present invention will be further disclosed as to the embodiments mentioned above.

<1>

45 **[0148]** A human hair fiber treatment agent comprised of one or more compositions for treating human hair fibers having free ends at both ends, wherein the human hair fiber treatment agent comprises the following components (A) to (C) in the formulation thereof:

50 (A): at least one formaldehyde derivative selected from the group consisting of formaldehyde, formaldehyde hydrate, glyoxylic acid, glyoxylic acid hydrate, glyoxylate, glyoxal, glyoxal hydrate, glutaraldehyde, and glutaraldehyde hydrate;
(B): phenol compound(s) having electron-donating group(s) on at least one of the meta positions and having hydrogen atom(s) on at least one of the ortho positions and the para position, with the proviso that the electron-donating group at the meta position optionally forms, together with a carbon atom attached thereto, a benzene ring optionally substituted with hydroxy group(s); and
55 (C): water.

<2>

[0149] The human hair fiber treatment agent according to <1>, wherein the component (A) preferably comprises one or more members selected from the group consisting of formaldehyde and glyoxylic acid.

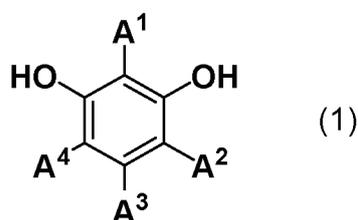
<3>

[0150] The human hair fiber treatment agent according to <1> or <2>, wherein the content of the component (A) is preferably 0.1% by mass or more, more preferably 1% by mass or more, further preferably 2.5% by mass or more, still further preferably 5% by mass or more, and preferably 60% by mass or less, more preferably 50% by mass or less, further preferably 40% by mass or less, further preferably 35% by mass or less, still further preferably 30% by mass or less, still further preferably 20% by mass or less, still further preferably 15% by mass or less.

<4>

[0151] The human hair fiber treatment agent according to any one of <1> to <3>, wherein the component (B) is preferably the following component (B1):

(B1): a resorcinol derivative represented by the formula (1) :



wherein

A¹ to A⁴ are the same or different and each represent a hydrogen atom, a hydroxy group, a halogen atom, a carboxyl group or a salt thereof, a sulfonic acid group or a salt thereof, a linear or branched alkyl group or alkenyl group having 1 to 6 carbon atoms, or a linear or branched alkoxy group or alkenyloxy group having 1 to 6 carbon atoms.

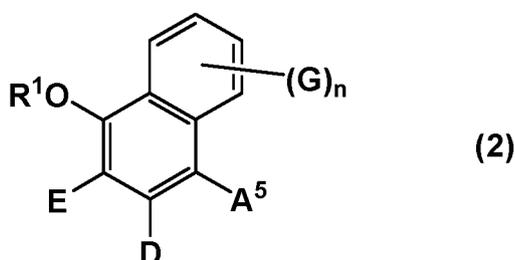
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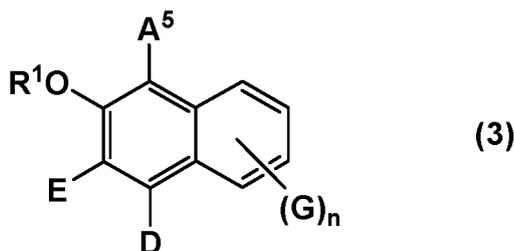
[0152] The human hair fiber treatment agent according to <4>, wherein the component (B1) is preferably selected from the group consisting of resorcinol, 2-methylresorcinol, 4-chlororesorcinol and pyrogallol.

<6>

[0153] The human hair fiber treatment agent according to any one of <1> to <3>, wherein the component (B) is preferably the following component (B2):

(B2): a naphthol derivative represented by the formula (2) or (3) :





wherein

R¹ represents a hydrogen atom or a methyl group;

A⁵ represents a hydrogen atom, a linear or branched alkyl group or alkenyl group having 1 to 12 carbon atoms, an optionally substituted aralkyl group or arylalkenyl group having 7 to 12 carbon atoms, a linear or branched alkoxy group or alkenyloxy group having 1 to 6 carbon atoms, a halogen atom or -CO-R² (wherein R² represents a linear or branched alkyl group or alkenyl group having 1 to 12 carbon atoms, an optionally substituted aralkyl group or arylalkenyl group having 7 to 12 carbon atoms or an optionally substituted aromatic hydrocarbon group having 6 to 12 carbon atoms);

D represents a hydrogen atom, a hydroxy group, a methyl group or a linear or branched alkoxy group or alkenyloxy group having 1 to 12 carbon atoms;

E represents a hydrogen atom, a hydroxy group, a linear or branched alkyl group or alkenyl group having 1 to 6 carbon atoms, or a linear or branched alkoxy group or alkenyloxy group having 1 to 6 carbon atoms;

G represents a hydroxy group, a linear or branched alkyl group or alkenyl group having 1 to 6 carbon atoms, or an alkoxy group having 1 to 6 carbon atoms; and n represents an integer from 0 to 2.

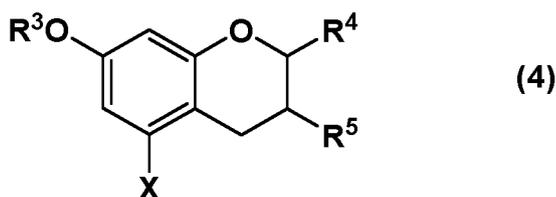
<7>

[0154] The human hair fiber treatment agent according to <6>, wherein the component (B2) is preferably selected from the group consisting of 1-naphthol, 2-naphthol, 3-methylnaphthalen-1-ol, naphthalene-1,5-diol and naphthalene-1,8-diol.

<8>

[0155] The human hair fiber treatment agent according to any one of <1> to <3>, wherein the component (B) is preferably the following component (B3):

(B3): a compound represented by the formula (4):



wherein

R³ represents a hydrogen atom or a methyl group;

X represents a hydrogen atom, a hydroxy group or a methoxy group;

R⁴ represents an aromatic hydrocarbon group optionally substituted by up to three hydroxy groups or methoxy groups and optionally forming a condensed ring with 1,3-dioxolane; and

R⁵ represents a hydroxy group, a methoxy group, an aromatic hydrocarbon group optionally substituted by up to three hydroxy groups or methoxy groups and optionally forming a condensed ring with 1,3-dioxolane, or an arylcarbonyloxy group or an aralkylcarbonyloxy group optionally substituted by up to three hydroxy groups or methoxy groups.

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<9>

5 **[0156]** The human hair fiber treatment agent according to <8>, wherein the component (B3) is preferably catechin, epicatechin, epigallocatechin, catechin gallate, epicatechin gallate or epigallocatechin gallate, more preferably one or more members selected from the group consisting of catechin, epigallocatechin and epigallocatechin gallate.

<10>

10 **[0157]** The human hair fiber treatment agent according to any one of <1> to <9>, wherein the content of the component (B) is preferably 0.1% by mass or more, more preferably 1% by mass or more, further preferably 2.5% by mass or more, still further preferably 5% by mass or more, and preferably 60% by mass or less, more preferably 50% by mass or less, further preferably 40% by mass or less, further preferably 35% by mass or less, still further preferably 30% by mass or less.

<11>

15 **[0158]** The human hair fiber treatment agent according to any one of <1> to <10>, wherein the molar ratio of the component (B) to the component (A), (B)/(A), is preferably 0.005 or more, more preferably 0.01 or more, further preferably 0.05 or more, still further preferably 0.1 or more, still further preferably 0.5 or more, and preferably less than 5, more preferably 4 or less, further preferably 3 or less, still further preferably 2 or less, still further preferably 1.8 or less.

<12>

20 **[0159]** The human hair fiber treatment agent according to any one of <1> to <11>, wherein the content of the component (C) is preferably 10% by mass or more, more preferably 20% by mass or more, further preferably 30% by mass or more, still further preferably 40% by mass or more, and preferably 99% by mass or less, more preferably 97% by mass or less, further preferably 95% by mass or less, still further preferably 90% by mass or less.

<13>

30 **[0160]** The human hair fiber treatment agent according to any one of <1> to <12>, wherein the human hair fiber treatment agent is preferably in a one-part form comprising the components (A) to (C) in a single composition.

<14>

35 **[0161]** The human hair fiber treatment agent according to <13>, wherein pH at 25°C is preferably 5.0 or lower, more preferably 4.8 or lower, further preferably 4.5 or lower, and preferably 0 or higher, more preferably 0.5 or higher, further preferably 1.0 or higher.

<15>

40 **[0162]** The human hair fiber treatment agent according to any one of <1> to <12>, wherein the human hair fiber treatment agent is preferably in a multiple-part form comprising a first part comprising the components (B) and (C), and a second part comprising the components (A) and (C).

<16>

45 **[0163]** The human hair fiber treatment agent according to <15>, wherein the content of the component (B) in the first part is preferably 0.1% by mass or more, more preferably 1% by mass or more, further preferably 2.5% by mass or more, still further preferably 5% by mass or more, and preferably 50% by mass or less, more preferably 40% by mass or less, further preferably 35% by mass or less, still further preferably 30% by mass or less.

<17>

55 **[0164]** The human hair fiber treatment agent according to <15> or <16>, wherein the content of the component (A) in the second part is preferably 0.1% by mass or more, more preferably 1% by mass or more, further preferably 2.5% by mass or more, still further preferably 5% by mass or more, and preferably 50% by mass or less, more preferably 40% by mass or less, further preferably 35% by mass or less, still further preferably 30% by mass or less.

<18>

[0165] The human hair fiber treatment agent according to any one of <15> to <17>, wherein pH at 25°C of each of the first part and the second part is preferably 6.0 or lower, more preferably 5.8 or lower, further preferably 5.5 or lower, and preferably 0 or higher, more preferably 0.5 or higher, further preferably 1.0 or higher, further preferably 2.0 or higher.

<19>

[0166] A method for treating human hair fibers, comprising the following step (i):

(i) the step of immersing human hair fibers having free ends at both ends in a human hair fiber treatment agent according to any of <1> to <18> under heating.

<20>

[0167] The method for treating human hair fibers according to <19>, wherein the bath ratio (human hair fiber treatment agent mass) / (human hair fiber mass) in immersing the human hair fibers in the human hair fiber treatment agent is preferably 2 or more, more preferably 3 or more, further preferably 5 or more, further preferably 10 or more, further preferably 20 or more, and preferably 500 or less, more preferably 250 or less, further preferably 100 or less.

<21>

[0168] The method for treating human hair fibers according to <19> or <20>, wherein the temperature of the human hair fiber treatment agent to immerse the human hair fibers is preferably 50°C or higher, more preferably 60°C or higher, further preferably 80°C or higher, and preferably lower than 100°C, more preferably 99°C or lower.

<22>

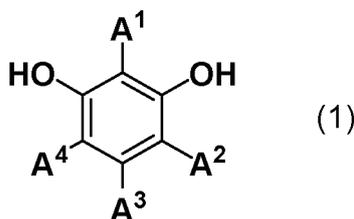
[0169] The method for treating human hair fibers according to any one of <19> to <21>, wherein the immersing time of the human hair fibers in the human hair fiber treatment agent is preferably 15 minutes or longer, more preferably 30 minutes or longer, further preferably 1 hour or longer, and preferably 48 hours or shorter, more preferably 24 hours or shorter, further preferably 12 hours or shorter.

<23>

[0170] The method for treating human hair fibers according to any one of <19> to <22>, preferably further comprising the following step (ii) after the step (i):

(ii) the step of immersing the human hair fibers in a surface finish agent (I) comprising the following components (B1) and (C):

(B1): a resorcinol derivative represented by the formula (1) :



wherein

A¹ to A⁴ are the same or different and each represent a hydrogen atom, a hydroxy group, a halogen atom, a carboxyl group or a salt thereof, a sulfonic acid group or a salt thereof, a linear or branched alkyl group or alkenyl group having 1 to 6 carbon atoms, or a linear or branched alkoxy group or alkenyloxy group having 1 to 6 carbon atoms; and (C): water.

<24>

[0171] The method for treating human hair fibers according to <23>, wherein the content of the component (B1) in the surface finish agent (I) is preferably 1% by mass or more, more preferably 2.5% by mass or more, further preferably 5% by mass or more, still further preferably 10% by mass or more, still further preferably 20% by mass or more, and preferably 95% by mass or less, more preferably 90% by mass or less, further preferably 88% by mass or less, still further preferably 85% by mass or less, still further preferably 80% by mass or less.

<25>

[0172] The method for treating human hair fibers according to <23> or <24>, wherein pH at 25°C of the surface finish agent (I) is preferably 7.0 or lower, more preferably 6.8 or lower, further preferably 6.5 or lower, and preferably 0 or higher, more preferably 0.5 or higher, further preferably 1.0 or higher.

<26>

[0173] The method for treating human hair fibers according to any one of <23> to <25>, wherein the bath ratio (the mass of the surface finish agent (I) / the mass of the human hair fibers treated in the step (i)) in immersing the human hair fibers in the surface finish agent (I) is preferably 2 or more, more preferably 3 or more, further preferably 5 or more, further preferably 10 or more, further preferably 20 or more, and preferably 500 or less, more preferably 250 or less, further preferably 100 or less.

<27>

[0174] The method for treating human hair fibers according to any one of <23> to <26>, wherein the immersing time of the human hair fibers in the surface finish agent (I) is preferably 1 hour or longer, more preferably 3 hours or longer, further preferably 6 hours or longer, still further preferably 24 hours or longer, and preferably 1 month or shorter, more preferably 2 weeks or shorter, further preferably 10 days or shorter, still further preferably 168 hours or shorter.

<28>

[0175] A method for producing a wig, comprising the step of treating human hair fibers for wigs by a method for treating human hair fibers according to any one of <19> to <27>.

<29>

[0176] A human hair fiber treatment agent comprised of one or more compositions for treating human hair fibers having free ends at both ends, wherein the human hair fiber treatment agent comprises the following components (A) to (C) and ethanol in the formulation thereof and has pH of 1.0 to 4.5:

- (A) 5 to 15% by mass of formaldehyde or glyoxal;
- (B) 10 to 20% by mass of resorcinol or 1-naphthol;
- (C) a balance of water; and

0 to 40% by mass of ethanol.

<30>

[0177] A method for treating human hair fibers, comprising the following step (i):

(i) the step of immersing human hair fibers having free ends at both ends in a human hair fiber treatment agent under heating of 90 to 99°C for 1 to 3 hours at a bath ratio of (human hair fiber treatment agent mass) / (human hair fiber mass) = 20 to 100, wherein the human hair fiber treatment agent comprised of one or more compositions, wherein the human hair fiber treatment agent comprises the following components (A) to (C) and ethanol in the formulation thereof and has pH of 1.0 to 4.5:

- (A) 5 to 15% by mass of formaldehyde or glyoxal;
- (B) 10 to 20% by mass of resorcinol or 1-naphthol;

(C) a balance of water; and

0 to 40% by mass of ethanol.

5 <31>

[0178] The method for treating human hair fibers according to <30>, preferably further comprising the following step (ii) after the step (i):

10 (ii) the step of immersing the human hair fibers treated in the step (i) in a surface finish agent (I) at a temperature of 20 to 90°C for 24 to 168 hours at a bath ratio of (the mass of the surface finish agent (I)) / (the mass of the human hair fibers treated in the step (i)) = 20 to 100, wherein the surface finish agent (I) comprises the following components (B1) and (C) and has pH of 1.0 to 6.5:

15 (B1) 20 to 80% by mass of resorcinol; and
(C) a balance of water.

<32>

20 **[0179]** A human hair fiber treatment agent for treating human hair fibers having free ends at both ends, the human hair fiber treatment agent being in a multiple-part form comprising the following first part and second part, wherein the human hair fiber treatment agent has pH of 2.0 to 5.0 when the first part and the second part are mixed in equal amounts:

the first part comprising the following components (B) and (C) and having pH of 2.0 to 5.5:

25 (B) 5 to 30% by mass of any one member selected from the group consisting of catechin, epigallocatechin and epigallocatechin gallate; and
(C) a balance of water; and

30 the second part comprising the following components (A) and (C) and ethanol and having pH of 1.0 to 4.5:

(A) 5 to 15% by mass of formaldehyde or glyoxal;
(C) a balance of water; and

35 0 to 40% by mass of ethanol.

<33>

[0180] A human hair fiber treatment agent kit comprising a first part and a second part according to <32> and further comprising a surface treatment agent comprising 20 to 80% by mass of resorcinol and water and having pH of 1 to 6.5.

40 <34>

[0181] A method for treating human hair fibers, comprising the following steps (i-1) and (i-2):

45 (i-1) the step of immersing human hair fibers having free ends at both ends in a first part under heating of 90 to 99°C for 1 to 3 hours at a bath ratio of (first part mass) / (human hair fiber mass) = 20 to 100, wherein the first part comprises the following components (B) and (C) and has pH of 2.0 to 5.5:

50 (B) 5 to 30% by mass of any one member selected from the group consisting of catechin, epigallocatechin, and epigallocatechin gallate; and
(C) a balance of water; and

55 (i-2) the step of immersing the human hair fibers treated in the step (i-1) in a second part under heating of 90 to 99°C for 1 to 3 hours at a bath ratio of (second part mass) / (the mass of the human hair fibers treated in the step (i-1)) = 20 to 100, wherein the second part comprises the following components (A) and (C) and ethanol and has pH of 1.0 to 4.5:

(A) 5 to 15% by mass of formaldehyde or glyoxal;

(C) a balance of water; and

0 to 40% by mass of ethanol.

5 <35>

[0182] The method for treating human hair fibers according to <34>, preferably further comprising the following step (ii) after the step (i):

10 (ii) the step of immersing the human hair fibers treated in the step (i) in a surface finish agent (I) at a temperature of 20 to 90°C for 24 to 168 hours at a bath ratio of (the mass of the surface finish agent (I)) / (the mass of the human hair fibers treated in the step (i)) = 20 to 100, wherein the surface finish agent (I) comprises the following components (B1) and (C) and has pH of 1.0 to 6.5:

15 (B1) 20 to 60% by mass of resorcinol; and
(C) a balance of water.

Examples

Examples 1 to 4 (Treatment with human hair fiber treatment agent in one-part form)

20 **[0183]** Each treatment agent shown in Table 1 was prepared and used in treatments given below to evaluate its shape sustainability, good surface feel, and durability. The results are also shown in Table 1. pH of each composition was measured directly from the prepared composition at room temperature (25°C) using a pH meter (manufactured by HORIBA, Ltd., F-52).

25 <Treatment with human hair fiber treatment agent>

[0184]

30 1. A 25 cm long tress consisting of 0.5 g of Caucasian hair (untreated hair / almost straight hair with very slight wave) was immersed in 40 g of the human hair fiber treatment agent in a container. The resulting container was hermetically sealed by covering the opening of the container with plastic wrap, and heated for 3 hours in an oven (Drying Oven Forced Convection System with Window Stainless; manufactured by AS ONE Corporation, SOFW-450) set to 90°C.

35 2. The container containing the tress was taken out of the oven and cooled to room temperature.

3. The tress was taken out of the container, rinsed under running tap water of 30°C for 30 seconds, washed with foams of shampoo for evaluation for 60 seconds, rinsed under running tap water of 30°C for 30 seconds, and slightly towel-dried. Then, the tress was dried with a warm-air dryer (manufactured by TESCOM & Co., Ltd., Nobby White NB3000). At this point in time, the tress maintained straight hair.

40 <Shape sustainability>

[0185]

45 1. The tress (straight hair) subjected to <Treatment with human hair fiber treatment agent> was wetted with tap water of 30°C for 30 seconds. Then, the wet tress was wrapped around a plastic rod having a diameter of 14 mm and fixed with a clip.

2. The whole rod was hermetically wrapped by covering with plastic wrap, and heated for 1 hour in an oven set to 90°C.

3. The tress was taken out of the oven and cooled to room temperature.

50 4. The tress was removed from the rod, rinsed under running tap water of 30°C for 30 seconds, and washed with foams of shampoo for evaluation for 60 seconds.

5. The tress was rinsed under running tap water of 30°C for 30 seconds, immersed in tap water of 30°C for 60 seconds at an infinite bath ratio, then gently pulled out of water by holding the root of the tress, and drained by light shaking.

55 6. The tress was left standing by hanging for 2 hours in a laboratory, dried, and combed. Then, the tress was hung and photographed from the side. On the basis of the photograph, the curvature radius (r) and curvature (1/r) of a most strongly curled part of the tress were calculated.

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(Evaluation criteria)

5 **[0186]** The curvature of untreated hair (straight hair) was defined as $1/r_0$, and the curvature measured by step 6 described above was defined as $1/r$. The rate (I) of increase in curvature (%) determined according to an expression given below was defined as shape sustainability (as the curled shape conferred by wrapping around a rod and heating is sustained as strong curl after hair wash, the value of I gets larger; the larger value of I means that the shape once conferred lasts longer, i.e., the shape sustainability is higher).

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$$I = [(1/r) / (1/r_0)] \times 100 [\%].$$

<Good surface feel>

15 **[0187]** For the evaluation of feel, the tress immediately after evaluation of <Shape sustainability> was used and evaluated for its smooth feel when touched by hands, by 5 dedicated panelists according to the criteria given below. A numeric value obtained by rounding off a mean from the 5 panelists was used as evaluation results.

(Evaluation criteria)

20 **[0188]**

5: very smooth hand feeling as compared with untreated hair.

25 4: smooth hand feeling as compared with untreated hair.

3: slightly smooth hand feeling as compared with untreated hair.

2: very slightly smooth hand feeling as compared with untreated hair.

30 1: the same hand feeling as in untreated hair.

<Durability>

35 **[0189]** For the evaluation of durability, the tress immediately after evaluation of <Shape sustainability> was used, and the tensile modulus of elasticity (Young's modulus) was used as an index for the durability of human hair fibers. The evaluation was conducted by the following steps.

40 1. Five human hair fibers were cut out from the root of the tress. A 3 cm human hair fiber specimen was collected from around the intermediate between the root and tip of each human hair fiber to obtain a total of ten 3 cm hair specimens. Each human hair fiber specimen was left in a room of 20°C and 60% RH for humidity condition for 24 hours.

45 2. The human hair fiber specimen was set in "MTT690 Automated Fiber Tensile Tester" manufactured by DIA-STRON limited. Automated measurement was started to determine the tensile modulus of elasticity (Young's modulus) in a wet state of the human hair fiber. The tensile modulus of elasticity (Young's modulus) shown in the tables was determined from a mean of the measurement results at N = 5. A higher numeric value means better strength as well as better durability.

<Formulation of shampoo for evaluation>

50 **[0190]**

Component	(% by mass)
Sodium laureth sulfate	15.5
Lauramide DEA	1.5
Sodium benzoate	0.5
EDTA-2Na	0.3
Phosphoric acid	amount necessary for pH adjustment to 7
Deionized water	balance

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(continued)

Component (% by mass)
Total 100.0

[Table 1]

Component (% by mass)			Example				Comparative Example
			1	2	3	4	1(*)
Human hair fiber treatment agent in one-part form	(A)	Formaldehyde	10	10	-	-	Not treated
		Glyoxylic acid	-	-	10	10	
	(B1)	Resorcinol	15	-	15	-	
	(B2)	1-Naphthol	-	15	-	15	
	(C)	Water	Balance	Balance	Balance	Balance	
	Others	Ethanol	-	40	-	40	
		Hydrochloric acid	(**)	(**)	-	-	
		Sodium hydroxide	-	-	(**)	(**)	
	Total		100	100	100	100	
pH (25°C)		2	2	2	2		
Evaluation	Shape sustainability (value of I)		625	714	500	385	100
	Good hair surface feel		2	3	2	3	1
	Durability (Young's modulus [GPa])		2.14	2.04	-	-	1.86
*: untreated hair							
**: amount for pH adjustment							

Examples 5 to 8 (Treatment with human hair fiber treatment agent in two-part form)

[0191] Each treatment agent shown in Table 2 was prepared and used in treatments given below to evaluate its shape sustainability, good surface feel, and durability according to the same method and criteria as in Examples 1 to 4. The results are also shown in Table 2. pH of each composition was measured directly from the prepared composition at room temperature (25°C) using a pH meter (manufactured by HORIBA, Ltd., F-52).

<Treatment with human hair fiber treatment agent>

[0192]

1. A 25 cm long tress consisting of 0.5 g of Caucasian straight hair (untreated hair) was immersed in 40 g of the first part of the human hair fiber treatment agent in a container. The resulting container was hermetically sealed by covering the opening of the container with plastic wrap, and heated for 2 hours in an oven set to 90°C.
2. The container containing the tress was taken out of the oven and cooled to room temperature.
3. The tress was taken out of the container, rinsed under running tap water of 30°C for 30 seconds, washed with foams of shampoo for evaluation for 60 seconds, rinsed under running tap water of 30°C for 30 seconds, and slightly towel-dried. Then, the tress was dried with a warm-air dryer (manufactured by TESCOM & Co., Ltd., Nobby White NB3000). At this point in time, the tress maintained straight hair.
4. Subsequently, the tress obtained in step 3 was immersed in 40 g of the second part of the human hair fiber

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treatment agent in a container. The resulting container was hermetically sealed by covering the opening of the container with plastic wrap, and heated for 1 hour in an oven set to 90°C.

5. The container containing the tress was taken out of the oven and cooled to room temperature.

5 6. The tress was taken out of the container, rinsed under running tap water of 30°C for 30 seconds, washed with foams of shampoo for evaluation for 60 seconds, rinsed under running tap water of 30°C for 30 seconds, and slightly towel-dried. Then, the tress was dried with a warm-air dryer (manufactured by TESCO M & Co., Ltd., Nobby White NB3000). At this point in time, the tress maintained straight hair.

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[Table 2]

Component (% by mass)	Example				Comparative Example	
	5	6	7	8		
Human hair fiber treatment agent in two-part form	(B3)	Catechin (*1)	12.5	12.5	12.5	1(*)
	(C)	Water	Balance	Balance	Balance	
	Others	Sodium hydroxide	(**)	(**)	(**)	
		Total	100	100	100	
		pH (25°C)	4	4	4	
		Formaldehyde	10	-	-	
	(A)	Glyoxylic acid	-	10	-	
		Glyoxal	-	-	10	
		Glutaraldehyde	-	-	10	
	(C)	Water	Balance	Balance	Balance	
Second part	Others	Phosphoric acid	2	2	2	
		Sodium hydroxide	(**)	(**)	(**)	
		Total	100	100	100	
		pH (25°C)	2	2	2	
Evaluation	Shape sustainability (value of I)		294	250	278	313
	Good hair surface feel		3	3	3	3
	Durability (Young's modulus [GPa])		2.16	-	-	-

*: untreated hair

** : amount for pH adjustment

* 1: green tea extracts (manufactured by Kao Corporation) (catechin concentration: total of all isomers)

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Examples 9 and 10 (Treatment with human hair fiber treatment agent in one-part form + treatment with surface finish agent (I))

[0193] Each treatment agent shown in Table 3 was prepared and used in treatments given below to evaluate its shape sustainability, good surface feel, and durability according to the same method and criteria as in Examples 1 to 4. The results are also shown in Table 3. pH of each composition was measured directly from the prepared composition at room temperature (25°C) using a pH meter (manufactured by HORIBA, Ltd., F-52).

<Treatment with human hair fiber treatment agent>

[0194]

1. A 25 cm long tress consisting of 0.5 g of Caucasian straight hair (untreated hair) was immersed in 40 g of the human hair fiber treatment agent in a container. The resulting container was hermetically sealed by covering the opening of the container with plastic wrap, and heated for 3 hours in an oven set to 90°C.
2. The container containing the tress was taken out of the oven and cooled to room temperature.
3. The tress was taken out of the container, rinsed under running tap water of 30°C for 30 seconds, washed with foams of shampoo for evaluation for 60 seconds, rinsed under running tap water of 30°C for 30 seconds, and slightly towel-dried. Then, the tress was dried with a warm-air dryer (manufactured by TESCOM & Co., Ltd., Nobby White NB3000). At this point in time, the tress maintained straight hair.
4. Subsequently, the tress obtained in step 3 was immersed in 40 g of the surface finish agent (I) in a container. The resulting container was hermetically sealed by covering the opening of the container with plastic wrap, and warmed for 72 hours in an oven set to 40°C.
5. The container containing the tress was taken out of the oven and cooled to room temperature.
6. The tress was taken out of the container, rinsed under running tap water of 30°C for 30 seconds, washed with foams of shampoo for evaluation for 60 seconds, rinsed under running tap water of 30°C for 30 seconds, and slightly towel-dried. Then, the tress was dried with a warm-air dryer (manufactured by TESCOM & Co., Ltd., Nobby White NB3000). At this point in time, the tress maintained straight hair.

[Table 3]

Component (% by mass)			Example				Comparative Example
			9	10	3	4	1(*)
Human hair fiber treatment agent in one-part	(A)	Glyoxylic acid	10	10	10	10	Not treated
	(B1)	Resorcinol	15	-	15	-	
	(B2)	1-Naphthol	-	15	-	15	
	(C)	Water	Balance	Balance	Balance	Balance	
	Others	Ethanol	-	40	-	40	
		Hydrochloric acid	(**)	(**)	(**)	(**)	
	Total		100	100	100	100	
pH (25°C)		2	2	2	2		
Surface finish agent (I)	(B1)	Resorcinol	40	40	Not treated	Not treated	
	(C)	Water	Balance	Balance			
	Others	Hydrochloric acid	(**)	(**)			
	Total		100	100			
	pH (25°)		2	2			

(continued)

Component (% by mass)		Example				Comparative Example
		9	10	3	4	
Evaluation	Shape sustainability (value of I)	500	385	500	385	100
	Good hair surface feel	4	5	2	3	1
	Durability (Young's modulus [GPa])	-	-	-	-	1.86
*: untreated hair **: amount for pH adjustment						

Examples 11 and 12 (Treatment with human hair fiber treatment agent in two-part form + treatment with surface finish agent (I))

[0195] Each treatment agent shown in Table 4 was prepared and used in treatments given below to evaluate its shape sustainability, good surface feel, and durability according to the same method and criteria as in Examples 1 to 4. The results are also shown in Table 4. pH of each composition was measured directly from the prepared composition at room temperature (25°C) using a pH meter (manufactured by HORIBA, Ltd., F-52).

<Treatment with human hair fiber treatment agent>

[0196]

1. A 25 cm long tress consisting of 0.5 g of Caucasian straight hair (untreated hair) was immersed in 40 g of the first part of the human hair fiber treatment agent in a container. The resulting container was hermetically sealed by covering the opening of the container with plastic wrap, and heated for 2 hours in an oven set to 90°C.

2. The container containing the tress was taken out of the oven and cooled to room temperature.

3. The tress was taken out of the container, rinsed under running tap water of 30°C for 30 seconds, washed with foams of shampoo for evaluation for 60 seconds, rinsed under running tap water of 30°C for 30 seconds, and slightly towel-dried. Then, the tress was dried with a warm-air dryer (manufactured by TESCOM & Co., Ltd., Nobby White NB3000). At this point in time, the tress maintained straight hair.

4. Subsequently, the tress obtained in step 3 was immersed in 40 g of the second part of the human hair fiber treatment agent in a container. The resulting container was hermetically sealed by covering the opening of the container with plastic wrap, and heated for 1 hour in an oven set to 90°C.

5. The container containing the tress was taken out of the oven and cooled to room temperature.

6. The tress was taken out of the container, rinsed under running tap water of 30°C for 30 seconds, washed with foams of shampoo for evaluation for 60 seconds, rinsed under running tap water of 30°C for 30 seconds, and slightly towel-dried. Then, the tress was dried with a warm-air dryer (manufactured by TESCOM & Co., Ltd., Nobby White NB3000). At this point in time, the tress maintained straight hair.

7. Subsequently, the tress obtained in step 6 was immersed in 40 g of the surface finish agent in a container. The resulting container was hermetically sealed by covering the opening of the container with plastic wrap, and warmed for 72 hours in an oven set to 40°C.

8. The container containing the tress was taken out of the oven and cooled to room temperature.

9. The tress was taken out of the container, rinsed under running tap water of 30°C for 30 seconds, washed with foams of shampoo for evaluation for 60 seconds, rinsed under running tap water of 30°C for 30 seconds, and slightly towel-dried. Then, the tress was dried with a warm-air dryer (manufactured by TESCOM & Co., Ltd., Nobby White NB3000). At this point in time, the tress maintained straight hair.

[Table 4]

Component (% by mass)	Example				Comparative Example	
	11	12	7	8		
Human hair fiber treatment agent in two-part form	(B3)	Catechin (*1)	12.5	12.5	12.5	1(*)
	(C)	Water	Balance	Balance	Balance	
	Others	Hydrochloric acid	(**)	(**)	(**)	
		Total	100	100	100	
		pH (25°C)	4	4	4	
	(A)	Formaldehyde	10	10	-	
		Glyoxylic acid	-	-	10	
	(C)	Water	Balance	Balance	Balance	
	Others	Phosphoric acid	2	2	-	
		Sodium hydroxide	(**)	(**)	(**)	
Surface finish agent (I)		Total	100	100	100	Not treated
		pH (25°C)	2	2	2	
	(B1)	Resorcinol	40	40		
	(C)	Water	Balance	Balance		
	Others	Hydrochloric acid	(**)	(**)	Not treated	
		Total	100	100	Not treated	
		pH (25°C)	2	2	2	
		Shape sustainability (value of I)	294	294	250	
		Good hair surface feel	5	3	3	
		Durability (Young's modulus [GPa])	-	-	-	
Evaluation		Shape sustainability (value of I)	294	294	250	100
		Good hair surface feel	5	3	3	1
		Durability (Young's modulus [GPa])	-	-	-	1.86

*: untreated hair

** : amount for pH adjustment

*1: green tea extracts (manufactured by Kao Corporation.) (catechin concentration: total of all isomers)

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[0197] Examples described above showed that human hair fibers excellent in shape sustainability and durability are obtained by the treatment of human hair fibers with the human hair fiber treatment agent of the present invention. Furthermore, it was confirmed that the obtained human hair fibers maintained natural appearance equivalent to hair of the human head.

Example 13 (Additional treatment with composition for cuticle removal)

[0198] Cuticle can be removed from the surface of human hair fibers by additionally performing the following steps before or after any treatment of Examples 1 to 12.

1. A 25 cm long tress consisting of 0.5 g of human hair fibers is immersed in 40 g of the composition for cuticle removal in a container and left standing for 2 hours.
2. The tress is taken out of the container, rinsed under running tap water of 30°C for 30 seconds, and slightly towel-dried. Then, the tress is dried with a warm-air dryer (manufactured by TESCOM & Co., Ltd., Nobby White NB3000).

Composition 1 for cuticle removal

[0199]

	(% by mass)
Sodium dichloroisocyanurate	5.00
Water	balance
pH:	5.0

Composition 2 for cuticle removal

[0200]

	(% by mass)
Sodium hypochlorite	5.00
Water	balance
pH:	5.0

Example 14 (Additional treatment with bleach composition)

[0201] Human hair fibers can be bleached by additionally performing the following steps before or after any treatment of Examples 1 to 12.

1. A 25 cm long tress consisting of 0.5 g of human hair fibers is immersed in 40 g of bleach composition 1 in a container and left standing for 2 hours.
2. The tress is taken out of the container, rinsed under running tap water of 30°C for 30 seconds, and slightly towel-dried. Then, the tress is dried with a warm-air dryer (manufactured by TESCOM & Co., Ltd., Nobby White NB3000).

Bleach composition 1

[0202]

	(% by mass)
Strong ammonia water (28% by mass)	2.70
Ammonium bicarbonate	4.70
Steartrimonium chloride (manufactured by Kao Corporation, QUARTAMIN 86W)	8.00
1-Hydroxyethylidene-1,1-diphosphonic acid (60% by mass) (DEQUEST 2010cs)	0.05
Hydrogen peroxide water (35% by mass)	10.90
Water	balance
pH:	9.5

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Example 15 (Additional treatment with hair dye composition)

[0203] Human hair fibers can be dyed by additionally performing the following steps before or after any treatment of Examples 1 to 12.

5 1. A 25 cm long tress consisting of 0.5 g of human hair fibers is immersed in 40 g of any of hair dye compositions 1 to 4 in a container (the hair dye compositions 1 and 4 were each used as a 1:1 mixture of the first part and the second part) and left standing for 2 hours.

10 2. The tress is taken out of the container, rinsed under running tap water of 30°C for 30 seconds, and slightly towel-dried. Then, the tress is dried with a warm-air dryer (manufactured by TESCOM & Co., Ltd., Nobby White NB3000).

Hair dye composition 1

[0204]

(First part)	(% by mass)
Cetearyl alcohol	10.8
Oleth-5	5.0
Oleic acid	2.5
Cocamide MEA	4.6
Sodium lauryl sulfate	1.7
Propylene glycol monostearate	0.6
Anhydrous sodium sulfite	0.5
Ammonia water (28% by mass)	6.5
Toluene-2,5-diamine sulfate	1.3
Resorcinol	0.5
Metaaminophenol	0.2
2,4-Diaminophenoxyethanol hydrochloride	0.02
Purified water	balance

(Second part)	(% by mass)
Hydrogen peroxide water (35% by mass)	17.1
Cetearyl alcohol	1.7
Sodium lauryl sulfate	0.2
Phosphoric acid	0.3
Salicylic acid	0.01
Purified water	balance

Hair dye composition 2

[0205]

	(% by mass)
Acid Red 33	0.5
Hydrochloric acid	amount for pH adjustment
Purified water pH: 4.0	balance

Hair dye composition 3

[0206]

	(% by mass)
Amodimethicone	1.50

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(continued)

		(% by mass)
5	Hydroxyethylcellulose	1.40
	Fragrance	0.30
	Acid Red 52	0.55
	Basic Red 51	0.28
	HC Red 3	0.01
10	Water	balance

Hair dye composition 4

[0207]

15	(First part)	(% by mass)
	Ammonium chloride	0.25
	Monoethanolamine	0.80
	Fragrance	0.30
20	p-Toluenediamine	0.55
	Resorcinol	0.28
	m-Phenylenediamine	0.01
	Water pH: 6.8	balance

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	(Second part)	(% by mass)
	Hydrogen peroxide	6.00
	Water	balance
30	pH:	3.4

Example 16 (Treatment with surface finish agent (II))

35 **[0208]** Each surface finish agent (II) shown in Tables 5 and 6 is applied according to the bath ratio shown in the tables to the human hair fibers after any treatment of Examples 1 to 12, and the human hair fibers can be thoroughly dried using a dryer without washing off the surface finish agent, thereby imparting hydrophobicity thereto and reducing friction. In addition, these effects can be sustained over a long period after the treatment.

40 **[0209]** pH in the tables is a value measured directly from each composition without dilution or the like at room temperature (25°C) using pH meter F-52 manufactured by HORIBA, Ltd.

[Table 5]

Content (% by mass; active component for all)		Surface finish agent (II)					
		1	2	3	4	5	6
45	(D) Epoxyaminosilane copolymer (*1)	1	1	1	1	1	1
	Ethanol	10	8	8	-	-	-
	Benzyl alcohol	-	2	-	-	-	-
50	Phenoxyethanol	-	-	2	-	-	-
	Ethyl lactate	-	-	-	10	-	-
	Diethanolamine	-	-	-	-	10	-
	Guanidine hydrochloride	-	-	-	-	-	10
55	Water (*2)	89	89	89	89	89	89
	Lactic acid	(*3)	(*3)	(*3)	(*3)	(*3)	(*3)

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(continued)

Content (% by mass; active component for all)	Surface finish agent (II)					
	1	2	3	4	5	6
Total amount	100	100	100	100	100	100
pH	3.5	3.5	3.5	3.5	3.5	3.5
Treatment bath ratio (surface finish agent (II) / human hair fibers)	0.5	0.5	0.5	0.5	0.5	0.5
*1: Silsoft CLX-E (manufactured by Momentive Performance Materials, Inc., polysilicone-29: 15% by mass). *2: containing water and dipropylene glycol derived from Silsoft CLX-E *3: amount for pH adjustment						

[Table 6]

Content (% by mass; active component for all)		Surface finish agent (II)							
		7	8	9	10	11	12	13	14
(D)	Epoxyaminosilane copolymer (*1)	0.1	0.5	5	1	1	1	1	1
	Ethanol	10	10	10	5	7	20	93.3	20
	Benzyl alcohol	-	-	-	-	-	-	-	2
	Phenoxyethanol	-	-	-	-	-	-	-	2
	Water (*2)	89.9	89.5	85	94	92	79	5.7	75
	Lactic acid	(*3)	(*3)	(*3)	(*3)	(*3)	(*3)	(*3)	(*3)
Total amount		100	100	100	100	100	100	100	100
pH		3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5
Treatment bath ratio (surface finish agent (II) / human hair fibers)		0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
*1: Silsoft CLX-E (manufactured by Momentive Performance Materials, Inc., polysilicone-29: 15% by mass). *2: containing water and dipropylene glycol derived from Silsoft CLX-E *3: amount for pH adjustment									

Claims

1. A human hair fiber treatment agent comprised of one or more compositions for treating human hair fibers having free ends at both ends, wherein the human hair fiber treatment agent comprises the following components (A) to (C) in the formulation thereof:

(A): at least one formaldehyde derivative selected from the group consisting of formaldehyde, formaldehyde hydrate, glyoxylic acid, glyoxylic acid hydrate, glyoxylate, glyoxal, glyoxal hydrate, glutaraldehyde, and glutaraldehyde hydrate;

(B): phenol compound(s) having electron-donating group(s) on at least one of the meta positions and having hydrogen atom(s) on at least one of the ortho positions and the para position, with the proviso that the electron-donating group at the meta position optionally forms, together with a carbon atom attached thereto, a benzene ring optionally substituted with hydroxy group(s); and

(C): water.

2. The human hair fiber treatment agent according to claim 1, wherein the content of the component (A) is 0.1% by mass or more and 60% by mass or less.

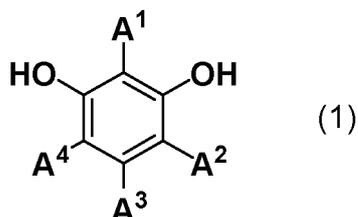
3. The human hair fiber treatment agent according to claim 1 or 2, wherein the content of the component (B) is 0.1% by mass or more and 60% by mass or less.

4. The human hair fiber treatment agent according to any one of claims 1 to 3, wherein the component (B) is the following component (B1):

(B1): a resorcinol derivative represented by the formula (1) :

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wherein

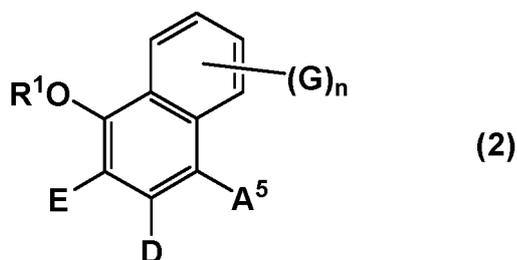
15 A¹ to A⁴ are the same or different and each represent a hydrogen atom, a hydroxy group, a halogen atom, a carboxyl group or a salt thereof, a sulfonic acid group or a salt thereof, a linear or branched alkyl group or alkenyl group having 1 to 6 carbon atoms, or a linear or branched alkoxy group or alkenyloxy group having 1 to 6 carbon atoms.

5. The human hair fiber treatment agent according to any one of claims 1 to 3, wherein the component (B) is preferably the following component (B2):

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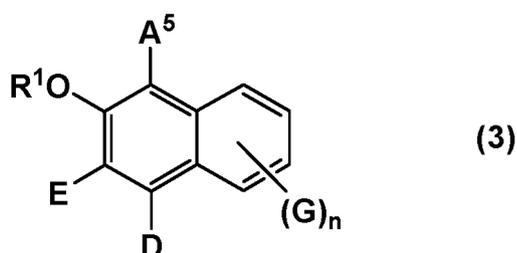
(B2): a naphthol derivative represented by the formula (2) or (3) :

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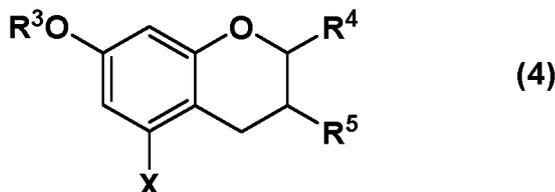
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wherein

45 R¹ represents a hydrogen atom or a methyl group;
A⁵ represents a hydrogen atom, a linear or branched alkyl group or alkenyl group having 1 to 12 carbon atoms, an optionally substituted aralkyl group or arylalkenyl group having 7 to 12 carbon atoms, a linear or branched alkoxy group or alkenyloxy group having 1 to 6 carbon atoms, a halogen atom or -CO-R² (wherein R² represents a linear or branched alkyl group or alkenyl group having 1 to 12 carbon atoms, an optionally substituted aralkyl group or arylalkenyl group having 7 to 12 carbon atoms or an optionally substituted aromatic hydrocarbon group having 6 to 12 carbon atoms);
50 D represents a hydrogen atom, a hydroxy group, a methyl group or a linear or branched alkoxy group or alkenyloxy group having 1 to 12 carbon atoms;
E represents a hydrogen atom, a hydroxy group, a linear or branched alkyl group or alkenyl group having 1 to 6 carbon atoms, or a linear or branched alkoxy group or alkenyloxy group having 1 to 6 carbon atoms;
55 G represents a hydroxy group, a linear or branched alkyl group or alkenyl group having 1 to 6 carbon atoms, or an alkoxy group having 1 to 6 carbon atoms; and n represents an integer from 0 to 2.

6. The human hair fiber treatment agent according to any one of claims 1 to 3, wherein the component (B) is preferably the following component (B3):

(B3): a compound represented by the formula (4):



wherein

15 R³ represents a hydrogen atom or a methyl group;

X represents a hydrogen atom, a hydroxy group or a methoxy group;

R⁴ represents an aromatic hydrocarbon group optionally substituted by up to three hydroxy groups or methoxy groups and optionally forming a condensed ring with 1,3-dioxolane; and

20 R⁵ represents a hydroxy group, a methoxy group, an aromatic hydrocarbon group optionally substituted by up to three hydroxy groups or methoxy groups and optionally forming a condensed ring with 1,3-dioxolane, or an arylcarbonyloxy group or an aralkylcarbonyloxy group optionally substituted by up to three hydroxy groups or methoxy groups.

7. A method for treating human hair fibers, comprising the following step (i):

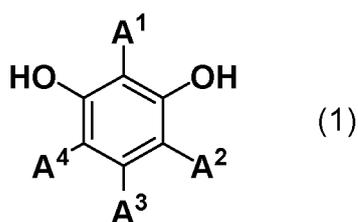
25 (i) the step of immersing human hair fibers having free ends at both ends in a human hair fiber treatment agent according to any of claims 1 to 6 under heating.

8. The method for treating human hair fibers according to claim 7, wherein the bath ratio (human hair fiber treatment agent mass) / (human hair fiber mass) in immersing the human hair fibers in the human hair fiber treatment agent is 2 or more.

9. The method for treating human hair fibers according to claim 7 or 8, further comprising the following step (ii) after the step (i):

35 (ii) the step of immersing the human hair fibers in a surface finish agent (I) comprising the following components (B1) and (C):

(B1): a resorcinol derivative represented by the formula (1) :



wherein

50 A¹ to A⁴ are the same or different and each represent a hydrogen atom, a hydroxy group, a halogen atom, a carboxyl group or a salt thereof, a sulfonic acid group or a salt thereof, a linear or branched alkyl group or alkenyl group having 1 to 6 carbon atoms, or a linear or branched alkoxy group or alkenyloxy group having 1 to 6 carbon atoms; and

(C): water.

10. A method for producing a wig, comprising the step of treating human hair fibers for wigs by a method for treating human hair fibers according to any one of claims 7 to 9.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2019/004787

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A. CLASSIFICATION OF SUBJECT MATTER
Int. Cl. A41G3/00 (2006.01) i, D06M13/11 (2006.01) i, D06M13/12 (2006.01) i,
D06M13/152 (2006.01) i

According to International Patent Classification (IPC) or to both national classification and IPC

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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
Int. Cl. A41G3/00, D06M13/11, D06M13/12, D06M13/152

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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Published examined utility model applications of Japan 1922-1996
Published unexamined utility model applications of Japan 1971-2019
Registered utility model specifications of Japan 1996-2019
Published registered utility model applications of Japan 1994-2019

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 2017-218397 A (KAO CORP.) 14 December 2017, paragraph [0008], [0071]-[0108], [0137], [0144], [0159] & WO 2017/207768 A1	1-10
Y	JP 2006-16310 A (KAO CORP.) 19 January 2006, paragraphs [0018], [0027]-[0030] (Family: none)	1-10

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Further documents are listed in the continuation of Box C. See patent family annex.

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* Special categories of cited documents:
 "A" document defining the general state of the art which is not considered to be of particular relevance
 "E" earlier application or patent but published on or after the international filing date
 "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
 "O" document referring to an oral disclosure, use, exhibition or other means
 "P" document published prior to the international filing date but later than the priority date claimed
 "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
 "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
 "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
 "&" document member of the same patent family

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Date of the actual completion of the international search 12.03.2019
Date of mailing of the international search report 26.03.2019

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REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- WO 2005037000 A [0006]
- JP 2007177370 A [0006]