Clarke, Modet & Cº
COLOMBIA

ESCOBAR - URIBE ASOCIADOS
Intellectual Property

As. 3062/PCT.

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
DIVISIÓN DE NUEVAS CREACIONES
E. S. D.

Ref.: Expediente No. **07-047.302**
Solicitud de Patente a nombre de
GIVAUDAN SA

Yo, **JIMENA ESCOBAR URIBE**, identificada como aparece al pie de mi firma, domiciliada en Bogotá, en la Carrera 11 No. 86-53, piso 6, obrando como apoderada de la sociedad **GIVAUDAN SA.,** con domicilio en Vernier, Suiza, solicitante de la patente de la referencia, pido a usted que se proceda a examinar si la invención, cuya patente se tramita en el expediente de la referencia, es patentable.

Adjunto al presente memorial el recibo No. 08 - 22,403 de 13 de marzo de 2008, por la suma de $420.000.oo, con lo cual queda cubierta la tasa oficial correspondiente a dicho estudio.

Señor Superintendente,

JIMENA ESCOBAR URIBE
C.C. 39.779.452 de Usaquen.
T.P. 68.228

3062. 92

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

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***** C O N S I G N A C I O N *****

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***** C O N C E P T O *****

CANT.	RENTISTICO	CONCEPTO	TOTAL CONCEPTO
1	50005 01 01 SOLICITUDES	112 PTE CAP I EXAMEN DE PATENTABILIDA	420,000.00
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2

Industria y Comercio
SUPERINTENDENCIA

93

DIVISIÓN DE NUEVAS CREACIONES

RADICACIÓN EXPEDIENTE No. 07-47302

EL EXTRACTO DE LA PRESENTE SOLICITUD DE **PATENTE DE INVENCIÓN**

FUE PUBLICADO EN LA GACETA DE PROPIEDAD INDUSTRIAL No **589** DE

FECHA **29 DE FEBRERO DE 2008** EL TÉRMINO HÁBIL SEÑALADO POR LA

LEY EXPIRA EL **03 DE JUNIO DE 2008**

NOTA: Dentro del plazo de los seis (6) meses siguientes a partir de la fecha de publicación en la gaceta de Propiedad Industrial, deberá solicitar y cancelar el examen de patentabilidad so pena de que la solicitud caiga en abandono, de acuerdo con lo establecido en el Artículo 44 de la Decisión 486/2000.

SE PRESENTARON OPOSICIONES POR PARTE DE TERCEROS.

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Raso et al. (43) **Pub. Date: May 1, 2003**

(54) **PROCESS FOR DISINFECTING A HARD-SURFACE WITH A COMPOSITION COMPRISING CINNAMON OIL AND/OR AN ACTIVE THEREOF**

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Publication Classification

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(52) **U.S. Cl.** **510/463**; 510/438

(57) **ABSTRACT**

The present invention relates to a process of disinfecting a hard-surface with a composition comprising cinnamon oil and/or an active thereof whereby disinfecting benefits are provided.

PROCESS FOR DISINFECTING A HARD-SURFACE WITH A COMPOSITION COMPRISING CINNAMON OIL AND/OR AN ACTIVE THEREOF

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] This is a continuation of International Application PCT/US01/11928 with an international filing date of Apr. 12, 2001, published in English under PCT Article 21(2) which claims benefit of European Application No. 00870072.6, filed Apr. 14, 2000.

TECHNICAL FIELD

[0002] The present invention relates to a process for disinfecting various hard-surfaces like walls, tiles, table tops, glass, bathroom surfaces, kitchen surfaces as well as dishes.

BACKGROUND

[0003] Hard-surfaces, like walls, tiles, table tops, glass as well as dishes, are prone to contamination with micro-organisms like bacteria, including Gram positive bacterial strains and Gram negative bacterial strains, viruses, and other more resistant micro-organisms like fungi. Hard-surfaces prone to contamination with micro-organisms can be found in various locations like : private households, for example, in kitchens and bathrooms; hospitals; restaurants; hotels; means of public transport; public bathes and pools; commercial and public laundries and the like.

[0004] Compositions for disinfecting hard-surfaces are well known in the art. Indeed, disinfecting compositions based on known disinfecting materials like bleach, quaternary ammonium compounds, essential oils or the like, provide acceptable disinfecting properties. For example, WO 97/25404 and WO 97/25106 describe disinfecting compositions based on essential oils and/or actives thereof in combination with hydrogen peroxide bleach.

[0005] Indeed, essential oils and/or actives thereof are known antimicrobial agents. Moreover, essential oils and/or actives thereof are, due to their availability from natural sources, preferable ingredients in hard-surface disinfectants. Indeed, it is well known from consumer research that consumers are looking for products comprising natural ingredients. However, it is well known that the overall disinfecting performance of the disinfecting compositions comprising essential oils and/or actives thereof used to disinfect hard-surfaces may still be further improved.

[0006] It is therefore an objective of the present invention to provide a process of disinfecting a hard-surface with a composition that delivers good overall disinfecting performance. In particular, it is an objective of the present invention to provide a process of disinfecting a hard-surface with a composition that delivers good disinfecting performance on Gram positive bacterial strains, like *Staphylococcus aureus* and Gram negative bacterial strains, like *Pseudomonas aeruginosa*, as well as on viruses and more resistant micro-organisms like fungi, under acidic, neutral and alkaline pH conditions.

[0007] It has now been found that the above objectives can be met by a process of disinfecting a hard-surface with a composition comprising cinnamon oil and/or an active thereof.

[0008] An advantage of the process as described herein is that said process provides an easy and fast way of disinfecting a hard-surface.

[0009] Another advantage of the present invention is that disinfection is provided on a broad range of bacterial strains including Gram positive bacterial strains, like *Staphylococcus aureus*, Gram negative bacterial strains, like *Pseudomonas aeruginosa*, viruses and more resistant micro-organisms like fungi, under acidic, neutral and alkaline pH conditions.

[0010] It is yet another advantage of the invention to provide a process of disinfecting a hard-surface with a composition that is mild to the skin as well as safe to the hard-surfaces treated therewith.

[0011] Another advantage of the present invention is that cinnamon oil and actives thereof are ingredients coming from natural sources and/or are present in nature.

BACKGROUND ART

[0012] The following documents are representative of the prior art available in the field of disinfecting compositions used on various surfaces.

[0013] EP 0 252 278 describes liquid mucous membrane disinfectants based on alcohol and hydrogen peroxide containing one or more carboxylic acids, nitrogen-containing organic compounds from the group of specific oligohexamethyl biguanides, and optionally microbiocidically active phenolic compounds as well as water.

[0014] WO 97/25404 and WO 97/25106 describe liquid disinfectant preparations for use on hard-surfaces comprising an essential oil or an active thereof.

SUMMARY OF THE INVENTION

[0015] The present invention encompasses a process of disinfecting hard-surfaces with a composition comprising cinnamon oil and/or an active thereof.

[0016] In a preferred embodiment, said composition comprises cinnamon acid, cinnamyl alcohol and/or cinnamyl aldehyde as an active of cinnamon oil.

[0017] In another preferred embodiment said composition is a liquid, preferably liquid aqueous, composition.

[0018] In another preferred embodiment said composition further comprises another essential oil and/or active thereof on top of the cinnamon oil and/or an active thereof, preferably an essential oil and/or active thereof selected from the group consisting of thymol and geraniol and mixtures thereof.

DETAILED DESCRIPTION OF THE INVENTION

[0019] Process of Disinfecting a Hard-Surface

[0020] The present invention encompasses a process of disinfecting a hard-surface with a composition, as defined herein, said process preferably comprising the step of applying said composition onto said surface.

[0021] The hard-surfaces to treat with the compositions herein are those typically found in houses like kitchens, bathrooms, e.g., tiles, walls, floors, chrome, glass, smooth vinyl, any plastic, plastified wood, table top, sinks, cooker

tops, dishes, sanitary fittings such as sinks, showers, shower curtains, wash basins, WCs and the like. Hard-surfaces also include household appliances including, but not limited to, refrigerators, freezers, washing machines, automatic dryers, ovens, microwave ovens, dishwashers and so on. Furthermore, hard-surfaces found in hospitals, restaurants, hotels, means of public transport, public bathes and pools, commercial and public laundries and the like are included herein.

[0022] In such a process a composition, as described herein, needs to be contacted with the hard-surfaces to be disinfected. Thus, the present invention also encompasses a process of disinfecting a hard-surface with a composition as described herein, wherein said process comprises the step of applying said composition to said hard-surface, preferably only infected portions thereof and optionally rinsing said hard-surface.

[0023] In the process of disinfecting hard-surfaces according to the present invention the compositions as described herein may be in liquid form and may be applied to the surface to be disinfected in their neat form or in their diluted form typically at a dilution level up to 100 times their weight of a suitable solvent, preferably water, preferably into 80 to 2 times their weight of a suitable solvent, preferably water, and more preferably 60 to 10 times their weight of a suitable solvent, preferably water.

[0024] In a preferred embodiment wherein the composition herein is incorporated onto a wipe, the process of disinfecting a hard-surface comprises the steps of contacting said wipe incorporation a composition as described herein with said hard-surface and preferably wiping said hard-surface with said wipe.

[0025] The Composition

[0026] The compositions of the present invention are preferably formulated as liquid compositions. Preferred compositions herein are aqueous compositions and therefore, preferably comprise water, more preferably in an amount of from 60% to 98%, even more preferably of from 80% to 97% and most preferably 85% to 97% by weight of the total composition.

[0027] The pH of the liquid compositions according to the present invention may typically be from 1 to 14.

[0028] In a preferred embodiment according to the present invention, the liquid compositions according to the present invention have an alkaline pH. More preferably, the pH of the liquid compositions herein, as is measured at 25° C., is at least, with increasing preference in the order given, 7.1, 7.5, 8.0, 8.5, 9.0, 9.25, 9.5 or 9.75. Independently, the pH of the liquid compositions herein, as is measured at 25° C., preferably is no more than, with increasing preference in the order given, 14.0, 13.5, 13.0, 12.5, 12.0, 11.5, 11.0, 10.75, 10.5, 10.25 or 10. Accordingly, the compositions herein may further comprise an acid, alkaline material and/or a buffering system to adjust and maintain pH as appropriate.

[0029] In another preferred embodiment, the liquid compositions according to the present invention have an acidic pH. More preferably, the pH of the liquid compositions herein, as is measured at 25° C., is at least, with increasing preference in the order given, 0.1, 0.5, 1.0, 1.5, 2, 2.5 or 3.0. Independently, the pH of the liquid compositions herein, as is measured at 25° C., preferably is no more than, with increasing preference in the order given, 6.9, 6.5, 6.0, 5.5, 5.0, 4.5, 4.0, 3.5 or 3.25. Accordingly, the compositions herein may further comprise an acid, alkaline material and/or a buffering system to adjust and maintain pH as appropriate. These preferred pH ranges also contribute to the stability of hydrogen peroxide, when present.

[0030] In still another preferred embodiment, the liquid compositions according to the present invention have a neutral pH.

[0031] Preferred acids herein to adjust the pH are organic or inorganic acids or mixtures thereof, these acids may be added on top of the organic acids and slats thereof as described herein below. Preferred organic acids are acetic acid, lactic acid or citric acid or a mixture thereof. Preferred inorganic acids are sulfuric acid or phosphoric acid or a mixture thereof. A particularly preferred acid to be used herein is an inorganic acid and most preferred is sulfuric acid.

[0032] Typical levels of such acids, when present, are of from 0.01% to 5.0%, preferably from 0.05% to 3.0%, and more preferably from 0.05% to 2.5% by weight of the total composition.

[0033] The alkaline material to be used herein to adjust the pH can be organic or inorganic bases. Suitable bases for use herein are the caustic alkalis, such as sodium hydroxide, potassium hydroxide and/or lithium hydroxide, and/or the alkali metal oxides such, as sodium and/or potassium oxide or mixtures thereof. A preferred base is a caustic alkali, more preferably sodium hydroxide and/or potassium hydroxide.

[0034] Other suitable bases include ammonia, ammonium carbonate and hydrogen carbonate.

[0035] Typical levels of such bases, are of from 0.01% to 5.0%, preferably from 0.05% to 3.0%, and more preferably from 0.05% to 2.5% by weight of the total composition.

[0036] Cinnamon Oil and/or an Active Thereof

[0037] As an essential ingredient the compositions according to the present invention comprise cinnamon oil and/or an active thereof.

[0038] By "active of cinnamon oil", it is meant herein any ingredient of cinnamon oil that exhibits antimicrobial activity.

[0039] Preferred actives of cinnamon oil are selected from the group consisting of cinnamic acid ($C_6H_5CH{=}CHCOOH$; 3-phenyl-2-propenoic acid); cinnamyl aldehyde ($C_6H_5CH{=}CHCHO$; 3-phenyl-2-propenal); cinnamyl alcohol ($C_6H_5CH{=}CHCH_2OH$; 3-phenyl-2-propen-1-ol); dehydro-cinnamyl aldehyde; amyl cinnamyl aldehyde; hexyl cinnamyl aldehyde; hexyl cinnamyl aldehyde; and α-n-butyl cinnamyl aldehyde; and mixtures thereof. More preferred actives of cinnamon oil are selected from the group consisting of: cinnamic acid ($C_6H_5CH{=}CHCOOH$; 3-phenyl-2-propenoic acid) and cinnamyl aldehyde ($C_6H_5CH{=}CHCHO$; 3-phenyl-2-propenal); and mixtures thereof. An even more preferred active of cinnamon oil is cinnamyl aldehyde ($C_6H_5CH{=}CHCHO$; 3-phenyl-2-propenal).

[0040] Cinnamon oil is commercially available from Sigma. Cinnamic acid is commercially available from Sigma. Cinnamyl alcohol is commercially available from

preferably from 0.006% to 10%, even more preferably from 0.006% to 8% and most preferably from 0.006% to 3%, by weight of the total composition.

[0058] In a preferred embodiment of the present invention, the compositions herein comprise a binary essential oil and/or active thereof system. By "binary essential oil and/or active thereof system" it is meant herein that in addition to the cinnamon oil and/or active thereof, the composition comprises one additional essential oil and/or active thereof (first additional essential oil and/or active). Preferably, said additional essential oil and/or active thereof is selected from the group consisting of eugenol, thymol and geraniol, more preferably selected from the group consisting of thymol and geraniol. Preferably, said binary essential oil and/or active thereof system comprises from 0.001% to 20%, preferably from 0.001% to 10%, by weight of the total composition of cinnamon oil and/or an active thereof; and from 0.001% to 10%, preferably from 0.001% to 5%, by weight of the total composition of the first additional essential oil and/or active thereof, preferably thymol or geraniol or eugenol, more preferably thymol or geraniol.

[0059] In another highly preferred embodiment of the present invention, the compositions herein comprise a ternary essential oil and/or active thereof system. By "ternary essential oil and/or active thereof system" it is meant herein that in addition to the cinnamon oil and/or active thereof, the composition comprises two additional essential oils and/or actives thereof (first and second additional essential oil and/or active). Preferably, said two additional essential oils and/or actives thereof are thymol and geraniol. Preferably, said ternary essential oil and/or active thereof system comprises from 0.001% to 20%, preferably from 0.001% to 10%, by weight of the total composition of cinnamon oil and/or an active thereof; from 0.001% to 10%, preferably from 0.001% to 5%, by weight of the total composition of the first additional essential oil and/or active thereof, preferably thymol; and from 0.001% to 10%, preferably from 0.001% to 5%, by weight of the total composition of the second additional essential oil and/or active thereof, preferably geraniol.

[0060] Indeed, it has been found that in the preferred embodiments herein, wherein the cinnamon oil and/or active thereof as described above, is combined with one or two other essential oils in a binary or a ternary essential oil system, the disinfecting properties of the compositions herein are even further increased compared to the compositions comprising cinnamon oil and/or an active thereof alone.

[0061] Organic Acid or a Salt Thereof

[0062] As an optional but highly preferred ingredient the compositions according to the present invention may comprise an organic acid or a salt thereof or a mixture thereof. Any organic acid or salt thereof known to those skilled in the art may be used herein. Organic acids and salts thereof, when present, contribute to the disinfection properties of the compositions described herein.

[0063] Suitable organic acids or salts thereof are selected from the group consisting of mono- and poly-carboxylic acids, percarboxylic acids and substituted carboxylic acids, and salts thereof, and mixtures thereof.

[0064] Suitable mono- and poly-carboxylic acids or salts thereof are selected from the group consisting of citric acid, lactic acid, ascorbic acid, isoascorbic acid, tartaric acid, formic acid, maleic acid, malic acid, malonic acid, propionic acid, acetic acid, dehydroacetic acid, benzoic acid, hydroxy benzoic acid, and salts thereof, and mixtures thereof.

[0065] Suitable percarboxylic acids or salts thereof are selected from the group consisting of peracetic acid, percarbonic acid, perboric acid, and salts thereof, and mixtures thereof.

[0066] Suitable substituted carboxylic acids or salts thereof are selected from the group consisting of aminoacids, halogenated carboxylic acids, and salts thereof, and mixtures thereof.

[0067] Preferred organic acids or salts thereof for use herein are selected from the group consisting of lactic acid, citric acid, and ascorbic acid and salts thereof and mixtures thereof. More preferred organic acids for use herein are selected from the group consisting of lactic acid and citric acid and salts thereof and mixtures thereof. An even more preferred organic acids for use herein is lactic acid or a salt thereof.

[0068] Suitable organic acids or salts thereof are commercially available from JBL, T&L, or Sigma. Lactic acid is commercially available from Sigma and Purac.

[0069] Typically the composition herein may comprise up to 20%, preferably from 0.1% to 10%, more preferably from 0.1% to 5%, even more preferably from 0.1% to 3%, by weight of the total composition of an organic acid or a salt thereof.

[0070] The Disinfecting Material

[0071] As an optional ingredient the compositions according to the present invention may comprise a disinfecting material or a mixture thereof, preferably an effective amount of a disinfecting material or a mixture thereof, in addition to the cinnamon oil and/or active thereof and the other essential oil and/or active thereof, when present.

[0072] By "disinfecting material", it is meant herein any known ingredient having the ability of reducing or even eliminating by killing the micro-organisms existing on a surface, not already listed herein above.

[0073] By "an effective amount of a disinfecting material", it is meant herein an amount sufficient to allow the disinfecting material to perform its action, i.e., to reduce the number of micro-organisms existing on a given surface. Depending on the disinfecting material used the amount used may be different. Typically, the compositions of the present invention comprise from 0.001% to 40%, preferably from 0.05% to 10% and more preferably from 0.1% to 5% by weight of the total composition of a disinfecting material.

[0074] Suitable disinfecting materials herein are all those known by those skilled in the art for the purpose of disinfecting and may include : bleaches like peroxygen bleaches and/or chlorine-type bleaches; quaternary ammonium compounds; phenolic compounds; aldehydes like glutaraldehyde and formaldehyde; glyoxal; parabens like ethyl paraben, propyl paraben, methyl paraben; biguanide antimicrobial agents; peroxy acids; and mixtures thereof.

[0075] Preferred disinfecting materials for use herein include a peroxygen bleach, or a biguanide antimicrobial agent or a mixture thereof.

[0076] A preferred disinfecting material for use herein is a peroxygen bleach or a mixture thereof. Preferred peroxygen bleach is hydrogen peroxide, or a water soluble source thereof, or mixtures thereof. Hydrogen peroxide is most preferred to be used herein.

[0077] The presence of the peroxygen bleach, especially hydrogen peroxide, in the compositions according to the present invention contribute to the disinfection properties of said compositions. Indeed, said peroxygen bleach may attack the vital function of the micro-organism cells, for example, it may inhibit the assembling of ribosomal units within the cytoplasm of the micro-organisms cells. Also the peroxygen bleach, like hydrogen peroxide, is an oxidizer that generates hydroxyl free radicals which attack proteins and nucleic acids. Furthermore, the presence of the peroxygen bleach, especially hydrogen peroxide, provides good stain removal benefits when used in any hard surface application.

[0078] As used herein a hydrogen peroxide source refers to any compound which produces hydrogen peroxide when said compound is in contact with water. Suitable water-soluble sources of hydrogen peroxide for use herein include percarbonates, persilicates, persulphates such as monopersulfate, perborates and peroxyacids such as diperoxydodecandioic acid (DPDA), magnesium perphthalic acid and mixtures thereof.

[0079] In addition, other classes of peroxides can be used as an alternative to hydrogen peroxide and sources thereof or in combination with hydrogen peroxide and sources thereof. Suitable classes include dialkylperoxides, diacylperoxides, preformed percarboxylic acids, organic and inorganic peroxides and/or hydroperoxides.

[0080] Typically, peroxygen bleach or a mixture thereof is present in the compositions according to the present invention at a level up to 20%, preferably from 0.1% to 15%, and more preferably from 0.5% to 10% by weight of the total composition.

[0081] Other suitable disinfecting materials for use herein include chlorine-type bleaches like hypochlorite.

[0082] Suitable quaternary ammonium compounds for use herein are quaternary ammonium compounds containing alkyl or substituted alkyl groups, alkyl amide and carboxylic acid groups, ether groups, unsaturated alkyl groups, and cyclic quaternary ammonium compounds, which can be chlorides, dichlorides, bromides, methylsulphates, chlorophenates, cylcohexylsulphamates or salts of the other acids. Among the possible cyclic quaternary ammonium compounds are the following:

[0083] alkylpyridinium chlorides and/or sulphates, the alkyl group being preferably cetyl, dodecyl or hexadecyl group;

[0084] alkylisoquinolyl chlorides and/or bromides, the alkyl group being preferably dodecyl group. Particularly suitable quaternary ammonium compounds for use herein include alkyl dimethyl benzyl ammonium chloride, octyl decyl dimethyl ammonium chloride, dioctyl dimethyl ammonium chloride, dodecyl dimethyl ammonium chloride, alkyl dimethyl ammonium saccharinate, cetylpyridinium and mixtures thereof.

[0085] Suitable phenolic compounds for use herein include o-phenyl-phenol, o-benzyl (p-chlorophenol), 4-tertamylphenol and mixtures thereof.

[0086] Suitable biguanide antimicrobial agents are characterised in comprising at least one, preferably 2 or more, biguanide moieties according to the following formula:

—NH—C(=NH)—NH—C(=NH)—NH—

[0087] Suitable and preferred biguanide antimicrobial agents are oligo- or poly alkylene biguanides or salts thereof or mixtures thereof. More preferred biguanide antimicrobial agents are oligo- or poly hexamethylene biguanides or salts thereof or mixtures thereof.

[0088] Examples of suitable biguanide antimicrobial agent are describe in EP 0 024 031, U.S. Pat. No. 2,684,924, U.S. Pat. No. 2,990,425, U.S. Pat. No. 3,468,898, U.S. Pat. No. 4,022,834, DE-OS-22 12 259 and DE-OS-26 27 548. Particularly suitable biguanide antimicrobial agents are selected from the group consisting of poly (hexamethylene biguanide) hydrochloride, 1,2-Bis-(N^5-p-chlorphenyl-N^1-biguanide)-ethane, 1,2-Bis-(N^5-p-nitrophenyl-N^1-biguanide)-ethane, 1,2-Bis-(N^5-p-hydroxyphenyl-N^1-biguanide)-ethane, 1,2-Bis-(N^5-p-chlorbenzyl-N^1-biguanide)-ethane, 1,2-Bis-(N^5-p-bromphenyl-N^5-hexyl-N^1-biguanide)-ethane, 1,2-Bis-(N^5-p-chlorphenyl-N^5-2-ethylphenyl-N^1-biguanide)-ethane, 1,2-Bis-(N^5-p-chlorphenyl-N^1-ethyl-N^1-biguanide)-ethane, 1,2-Bis-(N^5-p-methoxyphenyl-N^1-biguanide)-ethane, 1,2-Bis-(N^5-p-methylphenyl-N^1-biguanide)-ethane, 1,2-Bis-(N^5-3,5-dimethylphenyl-N^1-biguanide)-ethane, 1,2-Bis-(N^5-2,6-dichlorphenyl-N^1-biguanide)-ethane, 1,2-Bis-(N^5 -2,6-dimethylphenyl-N^1-biguanide)-ethane, 1,4-Bis-(N^5-p-chlorphenyl-N^1-biguanide)-butane, Bis-(N^5-p-chlorphenyl-N^1-biguanide)-methane, 1,3-Bis-(N^5-p-chlorphenyl-N^1-biguanide)-propane and 1,1'-hexamethylene-bis-[5-(4-chlorphenyl)-biguanide] and salts thereof, and mixtures thereof.

[0089] In a preferred embodiment according to the present invention said biguanide antimicrobial agents is a poly hexamethylene biguanide or salt thereof according to the following formula:

—[—$(CH_2)_3$—NH—C(=NH)—NH—C(=NH)—NH—$(CH_2)_3$—]—

[0090] wherein n is an integer selected from 1 to 50, preferably 1 to 20, more preferably 12. More preferably said biguanide antimicrobial agents is a salt of a poly hexamethylene biguanide according to the following formula:

—[—$(CH_2)_3$—NH—C(=NH)—NH—C(=NH)—NH—$(CH_2)_3$—]$_n$—.nHX

[0091] wherein n is an integer selected from 1 to 50, preferably 1 to 20, more preferably 12, and HX is salt component, preferably HCl.

[0092] A suitable poly hexamethylene biguanide or salt thereof is poly (hexamethylene biguanide) hydrochloride (PBG) wherein in the above formula n=12, commercially available under the trade name Vantocil IB® or Cosmocil CQ® from Avecia.

[0093] In another preferred embodiment according to the present invention said biguanide antimicrobial agents is 1,1'-hexamethylene-bis-[5-(p-chlorphenyl)-biguanide], commercially available under the trade name Chlrohexidine®.

[0094] Preferred biguanide antimicrobial agents according to the present invention are poly (hexamethylene biguanide) hydrochloride, preferably having a polymerization degree of 12, 1,1'-hexamethylene-bis-[5-(p-chlorphenyl)-biguanide] and mixtures thereof. A more preferred biguanide antimicrobial agents according to the present invention is poly (hexamethylene biguanide) hydrochloride, preferably having a polymerization degree of 12.

[0095] Typically the composition herein may comprise up to 20%, preferably from 0.01% to 20%, more preferably from 0.01% to 10%, even more preferably from 0.01% to 5%, by weight of the total composition of a biguanide antimicrobial agent.

[0096] Optional Ingredients

[0097] Surfactants

[0098] The compositions according to the present invention may further comprise a surfactant or mixtures thereof. Suitable surfactants to be used herein may be any surfactant known to those skilled in the art including anionic, nonionic, cationic, amphoteric and/or zwitterionic surfactants. Surfactants contribute to the cleaning performance of the disinfecting compositions of the present invention.

[0099] Particularly suitable anionic surfactants to be used herein include water soluble salts or acids of the formula $ROSO_3M$ wherein R is preferably a C_6-C_{24} hydrocarbyl, preferably an alkyl or hydroxyalkyl having a C_{10}-C_{20} alkyl component, more preferably a C_{12}-C_{18} alkyl or hydroxyalkyl, and M is H or a cation, e.g., an alkali metal cation (e.g., sodium, potassium, lithium), or ammonium or substituted ammonium (e.g., methyl-, dimethyl-, and trimethyl ammonium cations and quaternary ammonium cations, such as tetramethyl-ammonium and dimethyl piperdinium cations and quaternary ammonium cations derived from alkylamines such as ethylamine, diethylamine, triethylamine, and mixtures thereof, and the like).

[0100] Other suitable anionic surfactants to be used herein include alkyl-diphenyl-ether-sulphonates and alkyl-carboxylates. Other anionic surfactants can include salts (including, for example, sodium, potassium, ammonium, and substituted ammonium salts such as mono-, di- and triethanolamine salts) of soap, C_9-C_{20} linear alkylbenzenesulfonates, C_8-C_{22} primary or secondary alkanesulfonates, C_8-C_{24} olefinsulfonates, sulfonated polycarboxylic acids prepared by sulfonation of the pyrolyzed product of alkaline earth metal citrates, e.g., as described in British patent specification No. 1,082,179, C_8-C_{24} alkylpolyglycolethersulfates (containing up to 10 moles of ethylene oxide); alkyl ester sulfonates such as C_{14}-$_{16}$ methyl ester sulfonates; acyl glycerol sulfonates, fatty oleyl glycerol sulfates, alkyl phenol ethylene oxide ether sulfates, paraffin sulfonates, alkyl phosphates, isethionates such as the acyl isethionates, N-acyl taurates, alkyl succinamates and sulfosuccinates, monoesters of sulfosuccinate (especially saturated and unsaturated C_{12}-C_{18} monoesters) diesters of sulfosuccinate (especially saturated and unsaturated C_6-C_{14} diesters), acyl sarcosinates, sulfates of alkylpolysaccharides such as the sulfates of alkylpolyglucoside (the nonionic nonsulfated compounds being described below), branched primary alkyl sulfates, alkyl polyethoxy carboxylates such as those of the formula $RO(CH_2CH_2O)_kCH_2COO—M^+$ wherein R is a C_8-C_{22} alkyl, k is an integer from 0 to 10, and M is a soluble salt-forming cation. Resin acids and hydrogenated resin acids are also suitable, such as rosin, hydrogenated rosin, and resin acids and hydrogenated resin acids present in or derived from tall oil. Further examples are given in "Surface Active Agents and Detergents" (Vol. I and II by Schwartz, Perry and Berch). A variety of such surfactants are also generally disclosed in U.S. Pat. No. 3,929,678, issued Dec. 30, 1975 to Laughlin, et al. at Column 23, line 58 through Column 29, line 23 (herein incorporated by reference).

[0101] Preferred anionic surfactants for use in the compositions herein are the alkyl benzene sulfonates, alkyl sulfates, alkyl alkoxylated sulfates, paraffin sulfonates and mixtures thereof.

[0102] Suitable amphoteric surfactants to be used herein include amine oxides having the following formula $R_1R_2R_3NO$ wherein each of R_1, R_2 and R_3 is independently a saturated substituted or unsubstituted, linear or branched hydrocarbon chains of from 1 to 30 carbon atoms. Preferred amine oxide surfactants to be used according to the present invention are amine oxides having the following formula $R_1R_2R_3NO$ wherein R_1 is an hydrocarbon chain comprising from 1 to 30 carbon atoms, preferably from 6 to 20, more preferably from 8 to 16, most preferably from 8 to 12, and wherein R_2 and R_3 are independently substituted or unsubstituted, linear or branched hydrocarbon chains comprising from 1 to 4 carbon atoms, preferably from 1 to 3 carbon atoms, and more preferably are methyl groups. R_1 may be a saturated substituted or unsubstituted, linear or branched hydrocarbon chain.

[0103] Suitable amine oxides for use herein are for instance natural blend C_8-C_{10} amine oxides as well as C_{12}-C_{16} amine oxides commercially available from Hoechst.

[0104] Suitable zwitterionic surfactants to be used herein contain both cationic and anionic hydrophilic groups on the same molecule at a relatively wide range of pH's. The typical cationic group is a quaternary ammonium group, although other positively charged groups like phosphonium, imidazolium and sulfonium groups can be used. The typical anionic hydrophilic groups are carboxylates and sulfonates, although other groups like sulfates, phosphonates, and the like can be used. A generic formula for some zwitterionic surfactants to be used herein is

$$R_1—N^+(R_2)(R_3)R_4X^-$$

[0105] wherein R_1 is a hydrophobic group; R_2 and R_3 are each C_1-C_4 alkyl, hydroxy alkyl or other substituted alkyl group which can also be joined to form ring structures with the N; R_4 is a moiety joining the cationic nitrogen atom to the hydrophilic group and is typically an alkylene, hydroxy alkylene, or polyalkoxy group containing from 1 to 10 carbon atoms; and X is the hydrophilic group which is preferably a carboxylate or sulfonate group. Preferred hydrophobic groups R_1 are alkyl groups containing from 1 to 24, preferably less than 18, more preferably less than 16 carbon atoms. The hydrophobic group can be unsaturated and/or contain substituents and/or linking groups such as aryl groups, amido groups, ester groups and the like. In general, the simple alkyl groups are preferred for cost and stability reasons.

[0106] Highly preferred zwitterionic surfactants include betaine and sulphobetaine surfactants, derivatives thereof or mixtures thereof. Said betaine or sulphobetaine surfactants

of from 0.01% to 50% by weight of the total composition, preferably from 0.01% to 30% and more preferably from 0.05% to 20%.

[0118] Cleaning Test Method

[0119] Standard enamel plates are soiled by applying on them a grease/particulate matter and then baking them. The tested compositions are applied on a sponge that is placed onto a Gardner Machine. The Gardner machine measures the number of strokes needed to reach 95-99% clean plates. The performance is measured as such (i.e., undiluted) and upon dilution at 1.5% in water.

[0120] Chelating Agents

[0121] The compositions herein may further comprise a chelating agent as a preferred optional ingredient. Suitable chelating agents may be any of those known to those skilled in the art such as the ones selected from the group comprising phosphonate chelating agents, amino carboxylate chelating agents or other carboxylate chelating agents, or polyfunctionally-substituted aromatic chelating agents or mixtures thereof.

[0122] Such phosphonate chelating agents may include etidronic acid (1-hydroxyethylidene-bisphosphonic acid or HEDP) as well as amino phosphonate compounds, including amino alkylene poly (alkylene phosphonate), alkali metal ethane 1-hydroxy diphosphonates, nitrilo trimethylene phosphonates, ethylene diamine tetra methylene phosphonates, and diethylene triamine penta methylene phosphonates. The phosphonate compounds may be present either in their acid form or as salts of different cations on some or all of their acid functionalities. Preferred phosphonate chelating agents to be used herein are diethylene triamine penta methylene phosphonates. Such phosphonate chelants are commercially available from Monsanto under the trade name DEQUEST®.

[0123] Polyfunctionally-substituted aromatic chelating agents may also be useful in the compositions herein. See U.S. Pat. No. 3,812,044, issued May 21, 1974, to Connor et al. Preferred compounds of this type in acid form are dihydroxydisulfobenzenes such as 1,2-dihydroxy -3,5-disulfobenzene.

[0124] A preferred biodegradable chelating agent for use herein is ethylene diamine N,N'-disuccinic acid, or alkali metal, or alkaline earth, ammonium or substitutes ammonium salts thereof or mixtures thereof. Ethylenediamine N,N'-disuccinic acids, especially the (S,S) isomer have been extensively described in U.S. Pat. No. 4, 704, 233, Nov. 3, 1987 to Hartman and Perkins. Ethylenediamine N,N'-disuccinic acid is, for instance, commercially available under the tradename ssEDDS® from Palmer Research Laboratories.

[0125] Suitable amino carboxylate chelating agents useful herein include ethylene diamine tetra acetates, diethylene triamine pentaacetates, diethylene triamine pentaacetate (DTPA), N-hydroxyethylethylenediamine triacetates, nitrilotri-acetates, ethylenediamine tetrapropionates, triethylenetetraaminehexa-acetates, ethanoldiglycines, propylene diamine tetracetic acid (PDTA) and methyl glycine di-acetic acid (MGDA), both in their acid form, or in their alkali metal, ammonium, and substituted ammonium salt forms. Particularly suitable to be used herein are diethylene triamine penta acetic acid (DTPA), propylene diamine tetracetic acid (PDTA) which is, for instance, commercially available from BASF under the trade name Trilon FS® and methyl glycine di-acetic acid (MGDA).

[0126] Further carboxylate chelating agents to be used herein include malonic acid, salicylic acid, glycine, aspartic acid, glutamic acid, or mixtures thereof.

[0127] Said chelating agents, especially phosphonate chelating agents like diethylene triamine penta methylene phosphonates, are particularly preferred in the compositions according to the present invention as they have been found to further contribute to the disinfecting properties of the compositions herein.

[0128] Further chelating agents to be used herein include polymeric chelating agents, such as vinylpyrrolidone methacrylate copolymers, which are, for instance, commercially available from BASF under the trade name Luvitec VPMA 91W®.

[0129] Typically, the compositions according to the present invention comprise up to 5% by weight of the total composition of a chelating agent, or mixtures thereof, preferably from 0.002% to 3% by weight and more preferably from 0.002% to 1.5%.

[0130] Radical Scavengers

[0131] The compositions herein may further comprise a radical scavenger as a preferred optional ingredient. Suitable radical scavengers for use herein include the well-known substituted mono and di hydroxy benzenes and derivatives thereof, alkyl- and aryl carboxylates and mixtures thereof. Preferred radical scavengers for use herein include di-tert-butyl hydroxy toluene, hydroquinone, di-tert-butyl hydroquinone, mono-tert-butyl hydroquinone, tert-butyl-hydroxy anysole, benzoic acid, toluic acid, catechol, t-butyl catechol, 2-methoxy-phenol, 2-ethoxy-phenol, 4-allyl-catechol, 2-methoxy-4-(2-propenyl)phenol, benzylamine, 1,1,3-tris(2-methyl-4-hydroxy-5-t-butylphenyl) butane, as well as n-propyl-gallate. Highly preferred for use herein is di-tert-butyl hydroxy toluene, which is for example commercially available from SHELL under the trade name IONOL CP®.

[0132] Typically, the compositions according to the present invention comprise up to 5% by weight of the total composition of a radical scavenger, or mixtures thereof, preferably from 0.01% to 1.5% by weight and more preferably from 0.01% to 1%.

[0133] Solvents

[0134] When used, solvents will, advantageously, give an enhanced cleaning and disinfecting performance to the composition. Suitable solvents for incorporation in the compositions according to the present invention include all those known to those skilled in the art of hard-surfaces cleaner compositions. For example, suitable solvents for use herein include ethers and diethers having from 4 to 14 carbon atoms, preferably from 6 to 12 carbon atoms, and more preferably from 8 to 10 carbon atoms, glycols or alkoxylated glycols, glycol ethers and/or derivatives, polyols, alkoxylated aromatic alcohols, aromatic alcohols, aliphatic branched or linear alcohols, alkoxylated aliphatic branched or linear alcohols, terpenes, and mixtures thereof.

[0135] Suitable glycols for use herein are according to the formula HO—CR_1R_2—OH wherein R_1 and R_2 are indepen-

Ingredients	I	II	III	IV	V	VI
Water	(% w/w) to balance	(% w/w) to balance	(% w/w) to balance	(% w/w) to balance	(% w/w) to balance	(% w/w) to balance
Thymol	—	0.01	0.01	—	—	0.01
Geraniol	—	0.09	0.29	—	—	0.09
Cinnamon oil	—	—	—	—	0.2	0.2
Cinnamyl aldehyde	—	—	—	0.2	—	—
Perfume	0.0375	0.1	0.1	0.1	0.1	0.15
Ethanol	9.4	9.4	5.0	9.4	8.0	8.0
Silicone Dow AF	—	0.003	—	0.003	0.003	0.003
C_{12-14} amine oxide	—	0.2	0.2	—	0.1	—
C_{10} amine oxide	—	—	—	0.2	—	0.02
C_{9-10} EO10	1.0	0.2	—	0.8	0.1	—
C_{9-11} EO5	—	—	—	—	—	0.1
C_{12-14} Betaine sodium salt	0.25	—	—	—	—	—
2-Ethyl-Hexyl-Sulphate	0.75	0.1	0.1	0.15	0.05	0.05
Citric acid	1.5	0.75	1.5	1.0	—	—
Lactic acid	—	—	—	—	0.44	—
Na_2CO_3	—	0.1	—	—	0.06	—
NaOH	up to pH 10.0	up to pH 10.0	up to pH 10.0	up to pH 10.0	up to pH 10.0	up to pH 10.0
Disinfection performance	Lg. Red	Lg. Red	Lg. Red	Lg. Red	Lg. Red	Lg. Red
Gram negative bacterial strain (*P. aeruginosa*)	0.9	1.6	2.0	4.0	4.1	4.6
Gram positive bacterial strain (*S. aureus*)	1.5	1.7	3.7	4.5	4.5	4.8

[0176] The compositions I-VI described above were tested using test EN1276 as described in the section titled *Disinfecting test method*herein. Composition I-III are comparative examples. Compositions IV to VI are compositions according to the present invention.

[0177] The above results clearly show the disinfecting benefits both on gram negative and gram positive bacterial strains of a composition according to the present invention (compositions IV to VI), i.e., compositions comprising cinnamon oil or an active thereof, versus compositions comprising no essential oil or active thereof (composition I) or compositions comprising other essential oil or active thereof than cinnamon oil or an active thereof (composition II and m).

Examples

[0178] The following examples will further illustrate the present invention. The compositions are made by combining the listed ingredients in the listed proportions (weight % unless otherwise specified). Furthermore, the compositions I to L are comparative example compositions.

Ingredients	A	B	C	D	E	F
Water	(% w/w) to balance	(% w/w) to balance	(% w/w) to balance	(% w/w) to balance	(% w/w) to balance	(% w/w) to balance
Thymol	0.025	—	—	—	0.02	—
Geraniol	0.0375	—	0.3	—	—	0.1
Cinnamic acid	—	0.5	—	—	—	—
Cinnamyl aldehyde	—	—	0.3	—	—	0.2
Vantocil IB ™	—	—	0.3	—	0.1	—
Chlrohexidine ®	—	—	—	0.2	0.1	—
Perfume	0.0375	0.1	0.1	0.1	0.1	0.15
Ethanol	9.4	9.4	5.0	9.4	8.0	8.0
Silicone Dow AF	—	0.003	—	0.003	0.003	0.003
C_{12-14} amine oxide	—	0.2	0.2	—	0.1	—
C_{10} amine oxide	—	—	—	0.2	—	0.02

-continued

C_{9-10} EO10	1.0	0.2	—	0.8	0.1	—
C_{9-11} EO5	—	—	—	—	—	0.1
C_{12-14} Betaine sodium salt	0.25	—	—	—	—	—
2-Ethyl-hexyl-Sulphate	0.75	0.1	0.1	0.15	0.05	0.05
Citric acid	1.5	0.75	1.5	1.0	—	—
Lactic acid	—	—	—	—	0.44	—
Na_2CO_3	—	0.1	—	—	0.06	—
NaOH	—	0.45	—	—	0.2	—
pH	2.4	9.5	2.8	2.8	9.5	7.1

Ingredients	G	H	I	J	K	L
Water	(% w/w) to balance	(% w/w) to balance	(% w/w) to balance	(% w/w) to balance	(% w/w) to balance	(% w/w) to balance
Cinnamon oil	—	—	—	—	0.2	0.1
Cinnamic acid	0.1	—	0.2	—	—	—
Cinnamyl aldehyde	—	0.1	—	0.3	—	—
Thymol	0.025	—	—	—	—	—
Geraniol	0.0375	—	0.3	—	—	—
Vantocil LB ™	—	—	—	0.3	—	0.2
Chlrohexi-dine ®	—	—	—	—	0.5	—
Perfume	0.0375	0.1	0.1	0.1	0.1	0.15
Ethanol	9.4	9.4	5.0	9.4	8.0	8.0
Silicone Dow AF	—	0.003	—	0.003	0.003	0.003
C_{12-14} amine oxide	—	0.2	0.2	—	0.1	—
C_{10} amine oxide	—	—	—	0.2	—	0.02
C_{9-10} EO10	1.0	0.2	—	0.8	0.1	—
C_{9-11} EO5	—	—	—	—	—	0.1
C_{12-14} Betaine sodium salt	0.25	—	—	—	—	—
2-Ethyl-hexyl-Sulphate	0.75	0.1	0.1	0.15	0.05	0.05
Citric acid	1.5	0.75	1.5	1.0	—	—
Lactic acid	—	—	—	—	0.44	—
Na_2CO_3	—	0.1	—	—	0.06	—
NaOH	—	0.45	—	—	0.2	—
pH	2.4	9.5	2.8	2.8	9.5	7.1

[0179] Vantocil IB® is poly (hexamethylene biguanide) hydrochloride commercially available form ICI. Chlrohexidine® is 1,1'-hexamethylene-bis-[5-(p-chlorphenyl)-biguanide] commercially available from Sigma.

What is claimed is:

1. A method of disinfecting a hard-surface comprising the step of:

Contacting said surface with a composition comprising cinnamon oil and/or an active thereof wherein said cinnamon oil is selected from the group consisting of:

cinnamic acid ($C_6H_5CH{=}CHCOOH$; 3-phenyl-2-propenoic acid); cinnamyl aldehyde ($C_6H_5CH{=}CHCHO$; 3-phenyl-2-propenal); cinnamyl alcohol ($C_6H_5CH{=}CHCH_2OH$; 3-phenyl-2-propen-1-ol); dehydro-cinnamyl aldehyde; amyl cinnamyl aldehyde; hexyl cinnamyl aldehyde; hexyl cinnamyl aldehyde; (αn-butyl cinnamyl aldehyde and mixtures thereof

2. The method of claim 1, wherein said composition comprises up to 20% by weight of the total composition of cinnamon oil and/or an active thereof.

3. The method of claim 1, wherein said composition further comprises an essential oil or an active thereof or a mixture thereof in addition to said cinnamon oil or active thereof.

4. The method of claim 3, wherein said composition comprises up to 20% by weight of the total composition of said essential oil or an active thereof or a mixture thereof in addition to said cinnamon oil or active thereof.

5. The method of claim 3, wherein said essential oil is selected from the group consisting of oils obtained from thyme, lemongrass, citrus, lemons, oranges, anise, clove, aniseed, cinnamon, geranium, roses, mint, lavender, citronella, eucalyptus, peppermint, camphor, sandalwood, pine, vervain, rosmarin, fleagrass, ratanhiae, cedar and mixtures thereof; and

wherein said or an active thereof is selected from the group consisting of actives of essential oils consisting of thymol, eugenol, menthol, geraniol, verbenone, eucalyptol, pinocarvone, cedrol, anethol, cinnamic acid, cinnamic aldehyde, carvacrol, hinokitiol, berberine, ferulic acid, methyl salicylic acid, methyl salicylate terpineol and mixtures thereof.

6. The method of claim 1, wherein said composition comprises a binary essential oil comprising cinnamon oil

US006472361B1

(12) **United States Patent**
Leonard et al.

(10) **Patent No.: US 6,472,361 B1**
(45) **Date of Patent: Oct. 29, 2002**

(54) **LIQUID CLEANING COMPOSITION COMPRISING A SALT OF POLYCARBOXYLIC ACID**

(75) Inventors: **Isabelle Leonard**, Voroux-lez-Liers (BE); **Didier Dormal**, Aywaille (BE)

(73) Assignee: **Colgate-Palmolive Company**, New York, NY (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **10/119,256**

(22) Filed: **Apr. 9, 2002**

(51) **Int. Cl.**[7] **C11O 17/00**

(52) **U.S. Cl.** **510/421**; 510/422; 510/426; 510/477; 510/505

(58) **Field of Search** 510/421, 422, 510/426, 477, 505

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(74) *Attorney, Agent, or Firm*—Richard E. Nanfeldt

(57) **ABSTRACT**

An improvement is described in all purpose liquid cleaning composition which is especially effective in the removal of oily and greasy soil and contains, a C_1–C_4 alkanol, a perfume or essential oil, and water.

1 Claim, No Drawings

metal polyphosphates, alkali metal carbonates, alkali metal phosphonates and alkali metal citrates because these materials, if used in the instant composition, would cause the composition to have a high pH as well as leaving residue on the surface being cleaned.

The following examples illustrate liquid cleaning compositions of the described invention. Unless otherwise specified, all percentages are by weight. The exemplified compositions are illustrative only and do not limit the scope of the invention. Unless otherwise specified, the proportions in the examples and elsewhere in the specification are by weight.

EXAMPLE 1

The following compositions in wt. % were prepared by simple mixing at 25° C.:

	A	B
C9–C11 alcohol EO 2-3:1	1	2
Ethanol	0	1
C8–18 alcohol EO 7.5-8:1	4	4
Polycarboxylic acid sodium salt	0.25	0.5
Coconut fatty acid	0.2	0.5
Sodium cumene sulfonate 40%	4	4
MgSO4 7 H2O	0.5	1
Perfume	0.65	0.65
Water	Balance	Balance
Grease cutting		
Neat	84%	93%
Dilute Residue	88% Better than reference	99% Better than reference

What is claimed:

1. A liquid cleaning composition consisting of:

(a) 0.5% to 4% of a C_9–C_{11} alkanol EO2–3 nonionic surfactant;

(b) 2% to 7% of a C_8–C_{18} alkanol EO7–8.5 nonionic surfactant;

(c) 0.1% to 5% of a solubilizer selected from the group consisting of sodium xylene sulfonate and sodium cumene sulfonate and mixtures thereof;

(d) 0.05% to 2% of a fatty acid;

(e) 0.25% to 6% of magnesium inorganic salt;

(f) 0.1 to 5 wt. % of an essential oil or a perfume;

(g) 0.05% to 2.5% of an alkali metal salt of a polycarboxylic acid;

(h) a C1–C4 alkanol;

(g) a propylene glycol ether; and

(l) the balance being water.

* * * * *

GEL AROMATIC CONTAINING PERFUME IN HIGH STABILITY

Patent number: JP55015438 (A)

Publication date: 1980-02-02

Inventor(s): AIGAMI KOUJI; SUGANO MIKIO; SAKURAI AKIRA

Applicant(s): KAO CORP

Classification:

- international: ***A61L9/01;*** **A61L9/01;** (IPC1-7): A61K7/46

- european:

Application number: JP19780087949 19780719

Priority number(s): JP19780087949 19780719

Also published as:

JP57015905 (B)

JP1125846 (C)

Abstract of **JP 55015438 (A)**

PURPOSE:To prepare a gel aromatic composition which retains perfume compounded in an aqueous gel, in high stability, by adding a hydrotropic agent such as an alkali metal salt of benzoic acid, etc. to the composition. CONSTITUTION:0.1-5 wt% of one or more hydrotropic agents selected from alkali metal benzoate, alkali metal p-toluenesulfonate, and alkali metal xylenesulfonate, are added to an aromatic composition. It is especially effective to improve the stability of e.g. an ester perfumery such as linalyl acetate, benzyl acetate, etc., or an aldehyde perfumery such as cyclamen aldehyde, etc.

United States Patent [19]

Purohit et al.

[11] **Patent Number: 4,966,754**

[45] **Date of Patent: Oct. 30, 1990**

[54] **PRESERVATION OF COSMETIC COMPOSITIONS**

[75] Inventors: **Prakash C. Purohit; Lal G. Ramdeen,** Minneapolis, Minn.

[73] Assignee: **Aveda Corporation,** Minneapolis, Minn.

[21] Appl. No.: **229,234**

[22] Filed: **Aug. 8, 1988**

[51] **Int. Cl.**[5] **A61K 35/78;** A61K 7/06

[52] **U.S. Cl.** **424/195.1;** 514/844; 514/845; 514/846; 514/847

[58] **Field of Search** 424/195.1; 514/844, 514/845, 846, 847

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Primary Examiner—John W. Rollins
Assistant Examiner—W. Catchpole

[57] **ABSTRACT**

A cosmetic composition containing a dermatologically acceptable carrier or vehicle and certain essential oils as a preservative. The essential oil provides antimicrobial assurance against *Apergillus niger, Candida albicans, Staphylococcus aureus* and *Pseudomonas aeruginosa.* A method of preparing the preserved cosmetic composition is also described.

2 Claims, No Drawings

PRESERVATION OF COSMETIC COMPOSITIONS

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to the preservation of cosmetic compositions, which contain a dermatologically acceptable carrier or vehicle, against four microorganisms, namely *Aspergillus niger, Candida albicans, staphyloccus aureus* and *Pseudomonas aeruginosa*. The invention relates to the cosmetic compositions containing the preservative and a method and composition for incorporating the preservative into the cosmetic composition.

2. Description of Related Art

For years research has been conducted in efforts to produce a cosmetic preparation that also possesses microbiocidal properties, an antimicrobial cosmetic composition. Thus, cosmetics have been formulated with a variety of bactericides which are effective against microorganisms such as *Staphylococcus aureus*. Such bactericides or antimicrobial compounds are generally synthetic or compounds such as methyl or propyl paraben, Dowicil 200, or various quaternary compounds. Care must be taken that the antimicrobial agents are non toxic and do not irritate the skin to which the cosmetic is applied. Not only must the cosmetic vehicle or carrier be dermatologically acceptable, but also the antimicrobial agent, or preservative agent, should be dermatologically acceptable. An acceptable cosmetic should be preserved against, or contain an antimicrobial agent effective against, at least four groups of microorganisms, 1. Molds (*Aspergillus niger*), 2. Yeasts (*Candida albicans*), 3. Gram positives (*Staphylococcus aureus*) and 4. Gram negatives (*Pseudomonas aeruginosa*).

DESCRIPTION OF THE INVENTION

It has now been determined that certain of the essential oils are effective as antimicrobial agents against the four microorganisms, *Aspergillus niger, Candida albicans, Staphylococcus aureus* and *Pseudomonas aeruginosa*. There are hundreds of natural essential oils available. A good discussion of essential oils, their composition and production, can be found in Kirk-Othmer Encyclopedia of Chemical Technology, Third Edition, Volume 16, pages 307-329, copyright 1981, John Wiley & Sons, Inc., the disclosure of which is herein incorporated by reference. Applicants have found forty-two oils and their isomers which are useful against all four microorganisms and are accordingly effective for use as preservatives in cosmetic applications. The oils are useful alone, but a mixture of these provide the most effective preservative, or antimicrobial action The oils, by the emulsified composition of this invention, and the method of incorporation described herein, are easily and readily capable of incorporation into cosmetic compositions of virtually any cosmetic vehicle or carrier. Thus, the antimicrobial compositions of this invention are comprised of a dermatologically acceptable vehicle or carrier and as a preservative or antimicrobial agent, an effective amount of one or more of the forty-two essential oils, found to be effective against all four microorganisms noted above. These oils, hereinafter specified, are safe, mild and effective preservative agents compatible with conventional, known cosmetic ingredients.

Because of the nature of the essential oils, they cannot merely be added to the cosmetic vehicle or composition. A means of incorporating the oils into the cosmetic composition has been discovered, however. To incorporate the oils, an emulsion or solution thereof is first formed in a solubilizer or dispersant agent such as a polyoxyethylene sorbitan ester. Such materials are available commercially under the "POLYSORBATE" name, i.e. Polysorbate 20 or Polysorbate 80. Although perhaps not necessary to explain, Polysorbate 80 is described as a polyoxyethylene sorbitan mono-oleate, sometimes also described as sorbitan monooleatepolyoxyethylene. The compound is accordingly an oleate ester of sorbitol and its anhydrides condensed with polymers of ethylene oxide having approximately 20 oxyethylene units. The common fatty acid esters are the oleate noted above, laurate and stearate. The dispersant may be generally described as a polyoxyethylene sorbitan fatty acid ester in which the fatty acid contains 8 to 22 carbon atoms, more commonly 12-18 carbon atoms, and preferably 18 carbon atoms and the number of oxyethylene units will vary between 5-40, preferably on the order of 10 to 30 with about 20 being most preferred.

In the emulsion or solution of the sorbitan ester and the essential oil, the ratio by weight of ester to oil will vary from about 2:1 to about 6:1 with about 3:1 being preferred. Thus the antimicrobial oil will comprise from about 15% to about 35% by weight based on the total weight of oil and solubilizer. The emulsion is prepared by mixing the sorbitan ester and the oil under vigorous conditions as in a vortex mixer.

Many cosmetic preparations are emulsions of a water phase and an oil phase. In preparing such compositions the water phase and oil phase components are separately prepared and the two phases mixed at an elevated temperature generally above 40° C. to about 60°-90° C., preferably about 75° C., until a uniform mixture is achieved. The mixture is then cooled to a temperature of about 40° C. at which time the premix blend of sorbitan ester and essential oils is added slowly with mixing until a uniform cosmetic composition results. The temperatures will vary somewhat dependent on the particular cosmetic composition being prepared. In addition to emulsion types (oil-in-water or water-in-oil) the cosmetic composition may also consist of a single aqueous or aqueous-alcoholic phase.

By "cosmetic" as the term is used herein is intended to include all types of products which are applied in any manner directly to a person for cleansing or embellishment purposes. Such "cosmetic" compositions accordingly require a dermatologically acceptable carrier or vehicle.

Included within the scope of the term "cosmetic" are creams and lotions, such as cleansing creams, facial creams, shaving creams, hand lotions and balms, antipersperant lotions and creams, cleansing gels, bath oils and gelees, hair dressing gels, and creams and conditioners, make-up, suntan oils, lotions and creams and other cosmetic applications.

The essential oils and their isomers which have been discovered to posses antimicrobial properties against all four of the micro-organisms earlier noted are:

1. Armoise
2. Basil
3. Bay
4. Bois de Rose
5. Caraway
6. Cardamon
7. Cedarwood
8. Cinnamon Bark

-continued

9. Coriander
10. Clovebud
11. Estragon
12. Fennel Sweet
13. Geraniol from Palmarosa oil
14. Juniper Berries
15. Lemongrass 80% rectified
16. Linalool ex Bois de Rose
17. Marjoram
18. Methol Crystals laevo
19. Neroli Bigarde Petals
20. Oakmoss Empuree
21. Otto Rose Bulgarian
22. Rosemary
23. Thyme
24. Vanilla Resinold
25. Wintergreen
26. Eugenol
27. Linalyl Formate
28. Oil of Clove (Indonesian)
29. Oil of Pimento Berry (W. Indian)
30. Oil Bay (W. Indian Redist.)
31. Natural Bitter Almond Oil
32. Wine Fusel Oil American
33. Oil Pennyroyal Spanish
34. Oil Litsea Cubeba
35. Oil Lovage
36. Oil Buchu Leaves
37. Laevo Linalool
38. Hydroxycitronella ex Citradora
39. Hyperabsolute Styrax Incolore
40. Hydroxycitronella ex Citronella
41. Iso Eugenol
42. Terpeneol Super

While each of the above has been found to be effective, preferably a mixture of these has been found to be the most effective at the levels generally employed in a cosmetic application such as a lotion or cream. The blend of sorbitan ester and oil is preferably added or incorporated into the cosmetic composition at a level of about 8% or more by weight of the total cosmetic composition. At the preferred 3:1 weight ratio of sorbitan ester and oil, the anitmicrobial essential oil will be present at a level of about 2% by weight of the total cosmetic composition. Higher amounts of essential oil above 2% will also be effective. However, it is generally not necessary to exceed 3 or 5% thereof depending largely on the fragrance desired. A preferred blend of oils which provides optimum antimicrobial properties, providing a clean kill within 72 hours of all four microorganisms noted earlier hereinabove, is as follows:

Blend X	
Percent by Weight	Essential Oil
20.00	Linalool (ex. Bois de Rose)
5.00	Geraniol (ex. Palmarosa)
5.00	Lemongrass 80% rectified
20.00	Bois de Rose
20.00	Cedarwood Oil
5.00	Marjoram Oil
2.00	Cinnamon Bark Oil
2.00	Cardamon Oil
3.00	Neroli Bigarde Petals Oil
2.00	Vanilla Resinoid
5.00	Coriander Oil
2.00	Oakmoss empuree
2.00	Armoise Oil
5.00	Menthol Crystals laevo

-continued

Blend X	
Percent by Weight	Essential Oil
2.00	Rose absolute concrete (wax)

EXAMPLE 1

In use in cosmetic preparations or compositions it is generally necessary that the oils be diluted in order to incorporate them easily into the cosmetic composition. It was found that conventional disc methods were not satisfactory for testing for antimicrobial properties of the essential oils. Accordingly, a new method of identifying the essential oils for antimicrobial activity was developed which utilizes the novel blend of sorbitan ester and essential oil. This test which follows below is the one employed in evaluating an identifying the antimicrobial activity of the forty-two oils specified hereinabove.

ANTIMICROBIAL TEST

A mixture of 3 parts Polysorbate 20 and 1 part essential oil is prepared by mixing well in a Vortex shaker. A mixture of 0.2, 0.4 and 0.8 grams of this blend (equal to 0.5%, 1.0% and 2.0% respectively essential oil) is added to 10 milliliters of sterile nutrient broth in a test tube and vigorously vortexed. A liquid, 24-hour culture of each of the four microorganisms is introduced into separate test tubes containing the essential oil blend with sorbitan ester and the nutrient broth. One drop of liquid culture contained about 1 million microorganisms. The test tubes are vigorously shaken and incubated for 72 hours at 37° C. Growth of microorganisms is examined by a streak of the foregoing preparation on Trypticase soy agar (TSA) plates. Growth on the TSA plate indicated no inhibition and thus no antimicrobial action in the blend. No growth on the plate indicated inhibition and antimicrobial activity.

Using the preferred blend of essential oils, Blend X described earlier above, the results of the test above at the 0.5%, 1.0% and 2.0% levels of essential oil was observed, with the following results:

TABLE I

Level	a *A. niger*	b *C. albicans*	c *S. aureus*	d *Ps. aeruginosa*
0.5%	No growth	No growth	No growth	Growth
1.0%	No growth	No growth	No growth	Growth
2.0%	No growth	No growth	No growth	No growth

The results in Table I show a clean kill of all four microorganisms in 72 hours at the 2% level of essential oil.

EXAMPLE 2

In order to test the effectiveness of the essential oil, Blend X described earlier, in a conventional cosmetic composition or cleansing cream was prepared using the formulation and procedure below:

Formulation

Formulation	
Percentage by weight	

-continued

	Water Phase
42.44	Deionized Water
1.0	Guar hydroxypropyltrimonium chloride
2.0	Glycerin USP 96%
0.5	Potassium Stearate
3.5	Sodium lauriminodipropionate
0.5	Allantoin
3.33	Sodium Cocoyl Isethionate
	Oil Phase
3.0	Glyceryl Stearate
25.0	Caprylic/capric triglyceride
3.75	Stearic Acid
4.5	Cetyl Alcohol
1.12	PEG 5 soya sterol
0.38	Soy Sterol
1.5	Jojoba Oil
0.38	Tocopherol
	Antimicrobial Premix
2.0	Essential Oil (Blend X)
6.0	Polysorbate 20

Procedure

The guar gum is hydrated in water and then the remaining ingredients of the water phase are added in sequential order with constant mixing. The phase is heated to 75° C. In a separate kettle all the ingredients of the oil phase are added in sequential order and heated to 75° C. with constant mixing. The oil phase is added to the water phase and mixed while maintaining the temperature at 75° C. The mixing is continued until uniform. The cream is cooled to 40° C., and the premix blend of polysorbate 20 and Blend X is added to the cream slowly and mixed until completely uniform.

EXAMPLE 3

To test the cleansing cream of Example 2 having incorporated therein 2% of the essential oil, Blend X, 20 grams of cleansing cream was put in four 15×150 mm test tubes. Approximately one million microorganisms of each of the four types were introduced into each tube. One gram of the inoculated product was diluted with 9 milliliters of sterile diluent, plated and counts were observed. In five days counts were reduced from 1×10^6 to zero.

TABLE II

Time (hrs)	Count
0	1×10^6
48	8×10^2
120	0

TABLE II-continued

The foregoing illustrates that Blend X is an effective preservative in a cosmetic.

Similar results are obtained with other cosmetic applications and compositions such as lotions, creams other than cleansing creams, single or multiple phase systems, solutions or emulsions.

What is claimed:

1. A method of preserving a cosmetic composition from microbial action of the microorganisms *Aspergillus niger, Candida albicans, Staphylococcus aureus* and *Pseudomonas aeruginosa* comprising incorporating into said cosmetic composition an essential oil having antimicrobial activity against all four of said microorganisms, wherein said antimicrobial essential oil is a mixture of essential oils having the following composition by weight:

Percentage By Weight	Essential Oil
20.00	Linalool ex Bois de Rose
5.00	Geraniol ex Palmarosa
5.00	Lemongrass 80% rectified
20.00	Bois de Rose
20.00	Cedarwood Oil
5.00	Marjoram Oil
2.00	Cinnamon Bark Oil
2.00	Cardamon Oil
3.00	Neroli Bigarde Petals Oil
2.00	Vanilla Resinoid
5.00	Coriander Oil
2.00	Oakmoss empuree
2.00	Armoise Oil
5.00	Menthol Crystals laevo
2.00	Rose absolute concrete wax

and wherein said antimicrobial essential oil is incorporated into said cosmetic composition by first dissolving said antimicrobial essential oil in a polyoxyethylene sorbitan ester wherein the ratio by weight of said sorbitan ester to said antimicrobial essential oil is in the range of 2:1 to 6:1 and adding said resulting sorbitan ester and essential oil mixture to said cosmetic composition in an amount to provide said antimicrobial essential oil in said cosmetic composition of at least 2% by weight of said cosmetic composition.

2. A method as defined in claim 1 wherein said composition is a cosmetic cream or lotion.

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(54) Title: ANTIMICROBIAL PERFUMING COMPOSITIONS

(57) Abstract: The present invention describes perfumes and perfuming compositions having an antimicrobial activity and containing effective amounts of certain perfuming ingredients which have an antimicrobial activity as evaluated by the Microbial Reduction Test.

test in which the same bacterium has been submitted to the same testing sequence as above but without addition of a perfuming ingredient.

A perfuming ingredient is said to have successfully passed the test, i.e. is said to have an antimicrobial activity, when 100% of the respective bacteria have been eliminated.

According to the invention, the active molecules are defined as compounds which are active against 100% of the bacteria of two or three of the strains mentioned above. A non-limiting list of the compounds obeying the conditions of the present invention is given hereinbelow :

Decanal	Isoeugenal
10-Undecen-1-al	Nerol
Nonanal	Tetrahydrolinalool
4-Isopropylbenzaldehyde	Zestover [3]
4-Undecanolide	Intreleven aldehyde [4]
Citronellal	(2E,6Z)-2,6-nonadien-1-ol
Citronellol	γ-Dodecalactone
Cyclamen aldehyde	Floralozone [5]
Delphone [1]	Isobutylquinoleine
Dihydro eugenol	Lilial ® [6]
8-p-Menthanol	Mayol ® [7]
Dimetol [2]	Phenylhexanol
Geraniol	9-Decen-1-ol
3-(1,3-Benzodioxal-5-yl)-2-methylpropanal	

1) 2-Pentyl-1-cyclopentanone : origin : Firmenich SA, Geneva, Switzerland

2) 2,6-Dimethyl-2-heptanol : origin : Givaudan-Roure SA, Vernier, Switzerland

3) 2,4-Dimethyl-1-carbaldehyde : origin : Firmenich SA, Geneva, Switzerland

4) origin : International Flavors & Fragrances, USA

5) mixture of 3-(4-ethylphenyl)-2,2-dimethylpropanal + 3-(2-ethylphenyl)-2,2-dimethylpropanal : origin : International Flavors & Fragrances, USA

6) origin : Givaudan-Roure SA,Vernier, Switzerland

7) cis-7-p-menthanol : origin : Firmenich SA, Geneva, Switzerland

Therefore, in the context of the present invention, an active molecule is defined as a compound having an antimicrobial activity as tested by the Microbial Reduction Test when it falls under the list given here-above. The use of these compounds as antimicrobial agents is an object of the present invention.

Another object of the present invention are compositions having an antimicrobial action and containing an effective amount of active molecules as defined above. We have found that, in order to be effective, such compositions should contain at least about 30% by weight, of the above-defined active molecules, with respect to the total weight of the composition. The preferred compositions are those which contain about 50% by weight of active molecules, with respect to the total weight of the composition.

Another object of the invention consists of a perfuming composition or a perfumed product containing an antimicrobial composition as defined above.

It is hence possible to prepare perfumes and colognes having an antimicrobial activity, by using a composition comprising active molecules according to the present invention, thus providing antimicrobial perfuming compositions. The antimicrobial perfuming compositions as defined above can advantageously be used to perfume certain products, in particular consumer products in the field of household and body care.

As it will appear from the examples below, these products, thanks to the presence of the antimicrobial compositions incorporated therein, acquire an antimicrobial activity themselves.

The activity of the final products will of course depend on the amount of perfuming composition present. Non-limiting examples for this type of application include soaps, bath and shower gels, shampoos, deodorants and antiperspirants, cosmetic compositions, air-fresheners, liquid and solid detergents for the treatment of textiles, fabric softeners and all-purpose cleaners for household and also industrial use.

In these applications, the antimicrobial compositions can be used alone or in admixture with other perfuming ingredients, solvents or adjuvants of current use in

perfumery. The nature and the variety of these coingredients do not require a more detailed description here, which, moreover, would not be exhaustive, and the person skilled in the art will be able to choose the latter through its general knowledge and as a function of the nature of the product to be perfumed and of the desired olfactive effect. These perfuming ingredients belong to chemical classes as varied as alcohols, aldehydes, ketones, esters, ethers, acetates, nitriles, terpene hydrocarbons, sulfur- and nitrogen-containing heterocyclic compounds, as well as essential oils of natural or synthetic origin. A large number of these ingredients is moreover listed in reference textbooks such as the book of S. Arctander, Perfume and Flavor Chemicals, 1969, Montclair, New Jersey, USA, or its more recent versions, or in other works of similar nature.

The invention will now be illustrated in greater detail in the following examples.

Embodiments of the Invention

Example 1

An antimicrobial composition was prepared with the following ingredients :

Ingredients	Parts by weight
Benzyl acetate	500
Hexylcinnamic aldehyde	1000
(2E,6Z)-2,6-nonadien-1-ol*	5
Citronellol	500
Coumarine	300
γ-Dodecalactone	50
Lorysia ® [1]	1000
Heliotropine	200
Isobutylquinoleine	50
Lilial ®	2500
Mayol ®	1000

Phenethylol	400
x Phenylhexanol	1500
Polysantol ®	500
Total	9505

* in dipropylene glycol

x = active compound according to the present invention

1) 4-(1,1-dimethylethyl)-1-cyclohexyl acetate ; origin : Firmenich SA, Geneva, Switzerland

The composition thus prepared showed an antimicrobial activity of 100% against *Escherichia coli*, *Pseudomonas aeruginosa* and *Staphylococcus aureus*, as measured by the test according to the present invention.

Example 2

A perfuming composition was prepared by using the following ingredients :

Ingredients	Parts by weight
Benzyl acetate	500
Linalyl acetate	500
x Citronellol	300
x Cyclamen aldehyde	100
x γ-Dodecalactone	20
x Geraniol	200
Habanolide ® 1)	500
x Lilial ®	1000
x Mayol ®	300
Phenethylol	800
x Phenylhexanol	500

Benzyl salicylate	800
Terpineol	1000
Total	6520

x = active compound according to the present invention

1) mixture of pentadec-11-en-15-olide and pentadec-12-en-15-olide ; origin : Firmenich SA, Geneva, Switzerland

The composition thus prepared showed an antimicrobial activity of 100% against *Escherichia coli*, *Pseudomonas aeruginosa* and *Staphylococcus aureus*, as measured by the test according to the present invention.

Example 3

There was prepared a perfuming composition using the following ingredients :

Ingredients	Parts by weight
[X] Nonanal	10
Hexylcinnamic aldehyde	800
[X] Intreleven aldehyde	10
[X] 4-Undecanolide	10
[X] Citronellol	1500
[X] Geraniol	1000
Habanolide ® [1]	800
Iralia ® Total [2]	200
Isopentyrate [3]	400
Dorisyl [1]	800
Lyral (iso) [4]	500
[X] Phenylhexanol	1000
[X] Tetrahydrolinalool	1500
Total	8530

x = active compound according to the present invention

1) see Example 2

2) methylionone mixture ; origin : Firmenich SA, Geneva, Switzerland

3) 1,3-dimethyl-3-butenyl isobutyrate ; origin : Firmenich SA, Geneva, Switzerland

4) mixture of 4- and 3-(4-hydroxy-4-methylpentyl)-3-cyclohexene-1-carbaldehyde ; origin : International Flavors & Fragrances, USA

The composition thus prepared showed an antimicrobial activity of 100% against *Escherichia coli*, *Pseudomonas aeruginosa* and *Staphylococcus aureus*, as measured by the test according to the present invention.

Example 4

Activity of antimicrobial perfuming compositions in a fabric softener

The *in vitro* Bacterial Contact Time (BCT) test provides a measure of the efficacy with which a product solution, at a certain concentration, will kill a given type of bacteria in the solution. This test is described in the international patent application WO 98/16194, the content of which is here-included by reference.

General method :

Three different antimicrobial compositions of the invention containing different percentages of active compounds formulated at 1%, were tested in a fabric softener base prepared from the following ingredients :

Ingredients	Parts by weight
Stepantex® VS90 [1)]	16.5
$CaCl_2$ (10% aqueous solution)	0.2
Dye (1% aqueous solution)	0.3
Water	82.0
Total	99.0

1) origin : Stepan, France