

DESCRIPTION

Title of Invention

PERFUME COMPOSITION, THE METHOD OF IMPARTING FRAGRANCE
OR FLAVOR TO FOOD, BEVERAGE, COSMETICS, AND FOOD,
5 BEVERAGE, COSMETICS CONTAINING SAID PERFUME COMPOSITION

Technical Field

[0001] The present invention relates to a perfume composition that can be used as a food flavoring, cosmetic aroma or the like, and specifically, it relates
10 to a perfume composition having three compounds, 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one, combined in a specific proportion.

Background Art

15 [0002] Citral is an aromatic component with a strong lemon-like fresh quality, which is present in lemon, lime and grapefruit, and it is widely used as an important aromatic material for foods and beverages, and for cosmetic products.

20 [0003] On the other hand, citral is also known to decrease with heating and passage of time, producing an off-flavor (an undesirable off-taste or off-odor) (NPL 1). Under acidic conditions, in particular, citral in citral-containing products decreases in stages during
25 production, distribution and storage periods, its structure being altered by reactions such as oxidation, cyclization, hydration and isomerization that result in

reduced freshness and very strong deterioration odor, and tend to significantly lower the quality of the product.

[0004] In the past, many attempts have been made to
5 add antioxidants such as isoascorbic acid to prevent generation of the various deterioration odor-causing substances produced from citral (NPL 2), but while effective methods exist for inhibiting the generation of these deterioration odor-causing substances, it has not
10 been possible to prevent decomposition of citral itself, such that the freshness of citral is lost and, consequently, no fundamental solution has been found for maintaining the original fragrance properties of citral.

[0005] There are also known methods for inhibiting
15 structural changes in the compounds and preventing generation of deterioration odor-causing substances, by using highly stable citral analog instead of citral, that have fragrance properties similar to citral.

For example, 4,8-dimethyl-3,7-nonadien-2-ol,
20 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one are known as highly stable citral analogs. However, while the fragrances of these substances are similar to citral, they have characteristic aromas that include mixtures of different
25 types of fragrances, and such aromatic substances have been difficult to use in isolation.

[0006] Previous disclosures have included adding a

mixture of 4,8-dimethyl-3,7-nonadien-2-ol and 4,8-dimethyl-3,7-nonadien-2-one to yield a perfume composition having excellent olfactory properties and high color stability (PTL 1) or to yield a perfume composition having a natural, fresh, strong lemon-like fragrance (PTL 2), and combining the two compounds of 4,8-dimethyl-3,7-nonadien-2-ol and 4,8-dimethyl-3,7-nonadien-2-yl acetate with an alcohol derived from a terpene compound to impart a fresh, strong lemon-like taste and flavor and to yield a perfume composition having high stability over time in acidic beverages (PTL 3).

[0007] These publications disclose that addition of a mixture of 4,8-dimethyl-3,7-nonadien-2-ol and 4,8-dimethyl-3,7-nonadien-2-one, or combination of the two compounds of 4,8-dimethyl-3,7-nonadien-2-ol and 4,8-dimethyl-3,7-nonadien-2-yl acetate with an alcohol derived from a terpene compound allows production of a perfume composition with citral-like fragrance properties and high stability over time.

[0008] However, although a combination of the two compounds of 4,8-dimethyl-3,7-nonadien-2-ol and 4,8-dimethyl-3,7-nonadien-2-one has a fragrance more similar to citral than when they are used alone, the floral and cosmetic fragrance produced by their simultaneous use is perceived as being different quality, and it has therefore been inadequate in terms of the overall

natural quality.

[0009] In addition, even though combining the two compounds of 4,8-dimethyl-3,7-nonadien-2-ol and 4,8-dimethyl-3,7-nonadien-2-yl acetate with an alcohol

5 derived from a terpene compound also produces a fragrance similar to citral, this has been inadequate in that a lime-like fragrance begins to become noticeable with as deterioration takes place, producing alteration in the original fragrance, or in that it is

10 insufficiently reminiscent of lemon aroma, which is an important fragrance component of citral and a typical expression of flavor, and therefore further improvement has been ardently desired.

Citation List

15 Patent literature

[0010]

PTL 1: Japanese Patent Public Inspection No. 2003-517052

PTL 2: Japanese Unexamined Patent Application

Publication No. 2001-181670

20 PTL 3: European Patent Application Publication No. 1417896

Non-patent literature

[0011]

NPL 1 : Peter Schieberle and Werner Grosch; J. Agric.

25 Food Chem., 36, 797-800 (1988)

NPL 2 : Val E. Peacock and David W. Kuneman; J. Agric.

Food Chem., 33, 330-335 (1985)

Summary of Invention**Technical Problem**

[0012] With the increasingly diversifying preferences of consumers in recent years, there is high demand for development of products of greater variety. This trend is particularly notable in the food and beverage and cosmetic industries, where there is a strong demand for development of an abundant variety of foods and beverages and cosmetic products to satisfy consumer preferences. In addition to these demands, there are also new modern demands for aromatic components as one of ingredients of foods and beverages and cosmetic products.

In regard to aromatic substances, in particular, there is demand for an excellent effect of exhibiting fragrance and flavor with a more natural quality, for a fragrance and flavor with higher palatability, and for not only imparting satisfactory fragrance and flavor but also having higher stability without generating off-odors with passage of time.

It is an object of the present invention to provide a perfume composition that can impart a natural quality, to meet the trend toward consumer preferences for natural quality, and a highly palatability fragrance and flavor, and especially having a fragrance reminiscent of lemon aroma, with high stability and reduced generation of off-odor with passage of time, as

well as foods and beverages and cosmetic products comprising the perfume composition.

Solution to Problem

[0013] As a result of diligent research toward
5 solving the problems described above, the present inventors have found, surprisingly, that fragrance of lemon aroma is notably increased by combining three different analogs with different functional groups, namely an alcohol (4,8-dimethyl-3,7-nonadien-2-ol), an
10 ester (4,8-dimethyl-3,7-nonadien-2-yl acetate) and a ketone (4,8-dimethyl-3,7-nonadien-3-one). Also surprisingly, it was found that the ratio for the specific proportion of the three compounds is important, and further diligent research was conducted on the ratio.

15 As a result, it was found that the blending ratio of 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one is 0.02-0.4 for 4,8-dimethyl-3,7-nonadien-2-yl acetate and 0.04-0.8 for 4,8-dimethyl-3,7-nonadien-2-one
20 with respect to 1 for 4,8-dimethyl-3,7-nonadien-2-ol, and preferably 0.04-0.2 for 4,8-dimethyl-3,7-nonadien-2-yl acetate and 0.08-0.4 for 4,8-dimethyl-3,7-nonadien-2-one with respect to 1 for 4,8-dimethyl-3,7-nonadien-2-ol.

It was found, surprisingly, that by including
25 the three compounds in a formulated perfume in these specific proportions, it is possible to provide a perfume composition that maintains a strong lemon aroma

and freshness even after storage under acidic conditions, and that can impart a citral-like flavor with a natural quality essentially without generating off-odor, as well as foods and beverages and cosmetic products containing the aromatic compound, in contrast to citral-containing perfume compositions and the perfume compositions of PTLs 1-3, and the invention has been completed upon this finding.

[0014] It has not been previously disclosed that mixing the three compounds 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one can produce a perfume composition having high stability over time and having stronger citral-like lemon aroma and freshness than the prior art, and furthermore, it has been neither taught nor suggested that mixing these three compounds in a specific proportion can yield a perfume composition that maintains strong lemon aroma and freshness even after storage under acidic conditions, and that can impart a citral-like flavor with a natural quality essentially without generating off-odor.

[0015] (1) The invention is a perfume composition comprising the three compounds 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one, combined in a proportion of 0.02-0.4 for 4,8-dimethyl-3,7-nonadien-2-yl acetate and 0.04-0.8 for 4,8-dimethyl-3,7-nonadien-2-

one with respect to 1 for 4,8-dimethyl-3,7-nonadien-2-ol.

(2) The invention is also a food and beverage or a cosmetic product comprising a perfume composition according to (1) above.

5 **Advantageous Effects of Invention**

[0016] The perfume composition of the invention, when added to a food and beverage and a cosmetic product, can impart citral-like lemon aroma and freshness with a more natural quality than the prior art, and allows
 10 production of foods and beverages and cosmetic products having reduced generation of off-odor with passage of time, compared to citral-containing perfume compositions with similar fragrance properties.

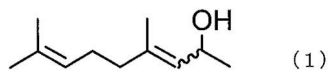
Description of Embodiments

15 [0017] The invention will now be explained in detail.

(1) Perfume composition

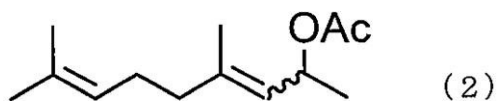
The invention is a perfume composition comprising three compounds:

20 4,8-dimethyl-3,7-nonadien-2-ol represented by formula (1):



(wherein the wave line bond indicates the cis form or trans form, or a mixture of the cis form and trans form in any proportion),

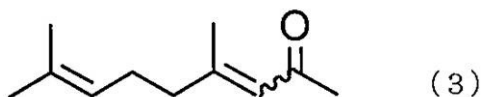
25 4,8-dimethyl-3,7-nonadien-2-yl acetate represented by formula (2):



(wherein the wave line bond indicates the cis form or trans form, or a mixture of the cis form and trans form in any proportion, and Ac represents an acetyl group),

5 and

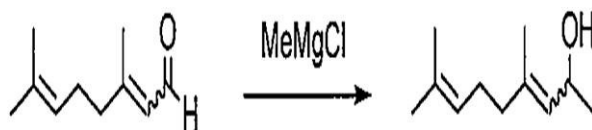
4,8-dimethyl-3,7-nonadien-2-one represented by formula (3):



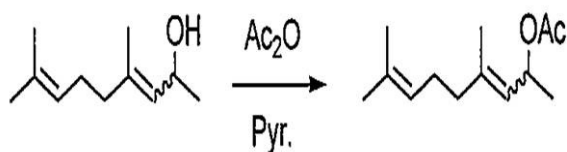
(wherein the wave line bond indicates the cis form or trans form, or a mixture of the cis form and trans form in any proportion).

[0018] The 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one to be used for the invention may be synthesized by methods described in any known publications.

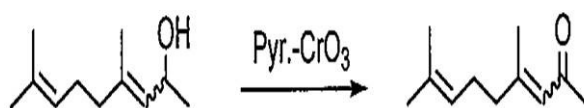
[0019] The 4,8-dimethyl-3,7-nonadien-2-ol can be obtained, for example, by treating citral with Grignard reaction with methylmagnesium chloride (MeMgCl), according to the following reaction formula (Agarwal et al.; Indian Perfum., 27,112-118(1983)).



[0020] The 4,8-dimethyl-3,7-nonadien-2-yl acetate can be obtained, for example, by acetylation of 4,8-dimethyl-3,7-nonadien-2-ol with acetic anhydride (Ac_2O) and pyridine (Pyr.), according to the following reaction formula (Agarwal et al.; Indian Perfum., 27, 112-118(1983)).



[0021] The 4,8-dimethyl-3,7-nonadien-2-one can be obtained, for example, by subjecting 4,8-dimethyl-3,7-nonadien-2-ol to Sarett oxidation with pyridine-chromium trioxide complex (Pyr.-CrO_3), according to the following reaction formula (Agarwal et al.; Indian Perfum., 27, 112-118(1983)).



[0022] However, the methods of synthesizing the 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one are not limited to these methods.

[0023] When the 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one, as active ingredients according to the

invention, are to be used in a perfume composition, the blending ratio of the three compounds may be 0.02-0.4 for 4,8-dimethyl-3,7-nonadien-2-yl acetate and 0.04-0.8 for 4,8-dimethyl-3,7-nonadien-2-one with respect to 1 for 4,8-dimethyl-3,7-nonadien-2-ol, and preferably 0.04-0.2 for 4,8-dimethyl-3,7-nonadien-2-yl acetate and 0.08-0.4 for 4,8-dimethyl-3,7-nonadien-2-one with respect to 1 for 4,8-dimethyl-3,7-nonadien-2-ol.

The amount of total addition with the three compounds will differ depending on the purpose and the type of perfume composition, but generally it may be in the range of 0.000000001 mass% (10 ppt) to 10 mass% and preferably 0.000001 mass% (10 ppb) to 5 mass%, with respect to the total perfume composition.

[0024] There are no particular restrictions on other components to be added to the perfume composition of the invention, and there may be used various aromatic materials conventionally used according to the application and purpose, which may include, specifically, aromatic materials known in the prior art such as aldehydes, alcohols and esters.

The blending method for the perfume composition is also not particularly restricted, and conventionally known blending methods may be mentioned, including methods of mixing each of the aromatic materials one at a time, and methods of adding a premixture of some of the aromatic materials.

[0025] (2) Application of perfume composition

The perfume composition of the invention can provide foods and beverages having citral-like fragrances and flavors with a natural quality, when
5 added to (a) beverages including tea drinks such as green tea, black tea and oolong tea, fruit beverages, alcoholic beverages, milk-based beverages and carbonated drinks; (b) frozen desserts such as ice cream, sherbet and ice candy; (c) fermented daily products such as
10 yogurt and cheese; (d) luxury products such as Japanese and Western confectioneries, baked confectioneries, jams, chewing gum, bread, cocoa, coffee, tea and tobacco; (e) desserts such as pudding, jelly, bavarois and mousse; (f) soups such as Japanese soups and Western soups; (g)
15 flavor seasonings; and (h) snack foods such as various types of instant beverages and foods.

[0026] In addition, the perfume composition of the invention may be generally used in a wide range of cosmetic products, including shampoos, hair creams,
20 pomades and other hair cosmetics, oshiroi, lipsticks and other cosmetic products, laundry detergents, disinfecting cleansers, indoor aromatic agents, and other health and hygienic materials, pharmaceuticals, and the like.

25 [0027] The amount of a perfume composition of the invention to be added to a food and beverage or cosmetic product differ depending on the type of food and

beverage or cosmetic product, but generally speaking, addition of the three compounds, 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one at a total

5 concentration of 0.000000000001 mass% (0.01 ppt) to 0.05 mass% in the food and beverage or cosmetic product can impart a citral-like fragrance and flavor with natural quality to the food and beverage or cosmetic product.

Examples

10 [0028] The invention will now be explained in greater detail through examples, with the understanding that the invention is in no way limited by the examples.

[0029]

[Aromatic synthesis examples]

15 (1) Synthesis of 4,8-dimethyl-3,7-nonadien-2-ol

After adding 100 ml of methylmagnesium chloride (3 M tetrahydrofuran solution) in 200 ml of tetrahydrofuran under a nitrogen stream and cooling to -5°C, 43.5 g of citral was added dropwise for a period of
20 30 minutes at -10°C to 0°C.

After reaction at -10°C to 0°C for 1 hour, the reaction mixture was added to 500 ml of an aqueous 10% ammonium chloride solution at 0°C to 10°C, and extraction was performed with 90 g of heptane. After
25 rinsing the heptane layer with saturated sodium bicarbonate water and sodium chloride solution, it was dried over anhydrous sodium sulfate and the heptane was

distilled off with a rotary evaporator to obtain 53 g of raw oil.

The raw oil was subjected to simple distillation to obtain 45 g of 4,8-dimethyl-3,7-nonadien-2-ol having a purity of 99.0% from gas chromatography analysis. (Boiling point: 59°C to 63°C/70 Pa)

[0030]

(2) Synthesis of 4,8-dimethyl-3,7-nonadien-2-yl acetate

10 After adding 5 g of sodium acetate to 25 g of 4,8-dimethyl-3,7-nonadien-2-ol and raising the temperature to 65°C, 59 g of acetic anhydride was added dropwise at 60°C to 70°C for a period of 10 minutes.

15 After reaction at 60°C to 70°C for 3 hours, the reaction mixture was cooled to 40°C, added to 150 g of water at 0°C to 10°C, and extracted with 50 g of heptane. After rinsing the heptane layer with water and 5% aqueous soda ash, it was dried over anhydrous sodium sulfate and the heptane was distilled off with a rotary
20 evaporator to obtain 31 g of raw oil.

The raw oil was subjected to simple distillation to obtain 30 g of 4,8-dimethyl-3,7-nonadien-2-yl acetate having a GC purity of 99.1%. (Boiling point: 71°C/80 Pa)

25 [0031]

(3) Synthesis of 4,8-dimethyl-3,7-nonadien-2-one

The 2 g of 4,8-dimethyl-3,7-nonadien-2-ol was

added 130 g of acetonitrile, 2.9 g of N-methylmorpholine-N-oxide and 4.6 g of molecular sieves 4A, and after cooling the mixture to -10°C, 125 mg of tetrapropylammonium perruthenate was added, and the
5 mixture was returned to room temperature while stirring for 2 hours.

The reaction mixture was added to 5% aqueous sodium thiosulfate, and was extracted with 50 g of heptane. After rinsing the heptane layer with 3%
10 aqueous hydrochloric acid and sodium chloride solution, it was dried over anhydrous sodium sulfate and the heptane was distilled off with a rotary evaporator to obtain 2.1 g of raw oil.

The raw oil was purified by silica gel column
15 chromatography (hexane:ethyl acetate = 19:1) and subjected to simple distillation to obtain 1.3 g of 4,8-dimethyl-3,7-nonadien-2-one having a purity of 99.7% from gas chromatography analysis. (Boiling point: 67°C/70 Pa)

20 [0032]

[Example 1 and Comparative Examples 1 to 4]
(Lemon flavor)

Perfume composition No.1 according to the invention having the following formulation was obtained
25 by a common method. There were also obtained, as Comparative Examples 1 to 4, perfume compositions No. 2 to 5, in which the mixing proportions of 4,8-dimethyl-

3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl

acetate and 4,8-dimethyl-3,7-nonadien-2-one were changed.

[0033]

[Table 1]

5

[Table 1] Perfume compositions (No.1-5)

10

Component name	Parts by weight				
	Example 1	Comp. Ex. 1	Comp. Ex. 2	Comp. Ex. 3	Comp. Ex. 4
Perfume composition	No.1	No.2	No.3	No.4	No.5
α -Pinene	0.1	0.1	0.1	0.1	0.1
β -Pinene	0.7	0.7	0.7	0.7	0.7
Myrcene	0.04	0.04	0.04	0.04	0.04
Limonene	2.8	2.8	2.8	2.8	2.8
1,8-Cineole	0.04	0.04	0.04	0.04	0.04
γ -Terpinene	0.4	0.4	0.4	0.4	0.4
Terpinolene	0.02	0.02	0.02	0.02	0.02
Octanal	0.08	0.08	0.08	0.08	0.08
Nonanal	0.13	0.13	0.13	0.13	0.13
Decanal	0.04	0.04	0.04	0.04	0.04
Citronellal	0.12	0.12	0.12	0.12	0.12
Linalool	0.14	0.14	0.14	0.14	0.14
4-Terpineol	0.05	0.05	0.05	0.05	0.05
α -Terpineol	0.3	0.3	0.3	0.3	0.3
Neryl acetate	0.18	0.18	0.18	0.18	0.18
Geranyl acetate	0.23	0.23	0.23	0.23	0.23
Peryl aldehyde	0.02	0.02	0.02	0.02	0.02
Nerol	0.05	0.05	0.05	0.05	0.05
Geraniol	0.05	0.05	0.05	0.05	0.05
Water	400	400	400	400	400
4,8-Dimethyl-3,7-nonadien-2-ol	0.55	0.55	0.55	0.55	0.55
4,8-Dimethyl-3,7-nonadien-2-	0.05	0.005	0.25	0.05	0.05

yl acetate					
4,8-Dimethyl-3,7-nonadien-2-one	0.1	0.1	0.1	0.01	0.5
Ethyl alcohol	remainder	remainder	remainder	remainder	remainder
Total	1000	1000	1000	1000	1000

[0034] The mixing proportions of 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one in the obtained perfume compositions No. 1 to 5) are shown in Table 2.

[0035]

[Table 2]

Table 2 Proportions of active ingredients added to perfume compositions (No.1-5)

Perfume composition	Example 1	Comp. Ex. 1	Comp. Ex. 2	Comp. Ex. 3	Comp. Ex. 4
	No.1	No.2	No.3	No.4	No.5
4,8-Dimethyl-3,7-nonadien-2-ol	1.0	1.0	1.0	1.0	1.0
4,8-Dimethyl-3,7-nonadien-2-yl acetate	0.09	0.009	0.45	0.09	0.09
4,8-Dimethyl-3,7-nonadien-2-one	0.18	0.18	0.18	0.02	0.91

[0036] Specifically, perfume composition No. 1 was a perfume composition with a blending ratio of 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one according to the invention, in the range of 0.02-0.4 for 4,8-dimethyl-3,7-nonadien-2-yl acetate and 0.04-0.8 for 4,8-

dimethyl-3,7-nonadien-2-one, with respect to 1 for 4,8-dimethyl-3,7-nonadien-2-ol.

[0037] On the other hand, perfume composition No. 2 was a perfume composition with a lower mixing proportion of 4,8-dimethyl-3,7-nonadien-2-yl acetate than the mixing proportion of the invention, perfume composition No. 3 was a perfume composition with a higher mixing proportion of 4,8-dimethyl-3,7-nonadien-2-yl acetate than the mixing proportion of the invention, perfume composition No. 4 was a perfume composition with a lower mixing proportion of 4,8-dimethyl-3,7-nonadien-2-one than the mixing proportion of the invention, and perfume composition No. 5 was a perfume composition with a higher mixing proportion of 4,8-dimethyl-3,7-nonadien-2-one than the mixing proportion of the invention.

[0038]

[Comparative Examples 5 to 8]

The following recipes, according to the inventions of PTLs 1 and 2, were used to obtain perfume composition No. 6 (Comparative Example 5), 2,4,8-trimethyl-7-nonen-2-ol was added instead of 4,8-dimethyl-3,7-nonadien-2-one in Example 1, according to the invention of PTL 3, were used to obtain perfume composition No. 7 (Comparative Example 6), citral was added instead of 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one in Example 1 to obtain perfume

composition No. 8 (Comparative Example 7), and ethyl alcohol was added instead of 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one in Example 1 to obtain

5 perfume composition No. 9 (Comparative Example 8).

[0039]

[Table 3]

Table 3 Perfume composition (No. 6-9)

10

Component name	Parts by weight			
	Comp. Ex. 5	Comp. Ex. 6	Comp. Ex. 7	Comp. Ex. 8
Perfume composition	No.6	No.7	No.8	No.9
α -Pinene	0.1	0.1	0.1	0.1
β -Pinene	0.7	0.7	0.7	0.7
Myrcene	0.04	0.04	0.04	0.04
Limonene	2.8	2.8	2.8	2.8
1,8-Cineole	0.04	0.04	0.04	0.04
γ -Terpinene	0.4	0.4	0.4	0.4
Terpinolene	0.02	0.02	0.02	0.02
Octanal	0.08	0.08	0.08	0.08
Nonanal	0.13	0.13	0.13	0.13
Decanal	0.04	0.04	0.04	0.04
Citronellal	0.12	0.12	0.12	0.12
Linalool	0.14	0.14	0.14	0.14
4-Terpineol	0.05	0.05	0.05	0.05
α -Terpineol	0.3	0.3	0.3	0.3
Neryl acetate	0.18	0.18	0.18	0.18
Geranyl acetate	0.23	0.23	0.23	0.23
Peryl aldehyde	0.02	0.02	0.02	0.02
Nerol	0.05	0.05	0.05	0.05
Geraniol	0.05	0.05	0.05	0.05
Water	400	400	400	400
4,8-Dimethyl-3,7-nonadien-2-ol	0.1	0.23	—	—
4,8-Dimethyl-3,7-nonadien-2-yl acetate	—	0.23	—	—
4,8-Dimethyl-3,7-	0.3	—	—	—

nonadien-2-one				
2,4,8-Trimethyl-7-nonen-2-ol	-	0.01	-	-
Citral	-	-	1	-
Ethyl alcohol	remainder	remainder	remainder	remainder
Total	1000	1000	1000	1000

[0040] The fragrances and flavors of the perfume compositions of Example 1 and Comparative Examples 1 to 8 (Nos. 1 to 9) were evaluated by a panel of 10 experts based on the evaluation criteria listed below, the average values are shown in Table 4, with the comments obtained during the evaluation being listed in Table 5.

[0041]

Fragrance and flavor evaluation criteria:

10 Very strongly noticeable: 5

Strongly noticeable: 4

Noticeable: 3

Slightly noticeable: 2

Very weakly noticeable: 1

15 [0042]

[Table 4]

Table 4 Average values of evaluation results for fragrances and flavors of perfume compositions of Example 1 and Comparative Examples 1-8 (Nos. 1-9)

20

	Perfume composition	Lemon flavor	Natural quality	Freshness	Remarks
Example 1	No.1	3.8	3.6	3.8	
Comp. Ex. 1	No.2	2.4	2.0	3.0	
Comp. Ex. 2	No.3	1.8	1.3	1.9	
Comp. Ex. 3	No.4	3.3	1.8	3.2	

Comp. Ex. 4	No.5	1.9	1.3	2.0	
Comp. Ex. 5	No.6	2.1	1.4	2.4	Perfume compositions of PTLs 1, 2
Comp. Ex. 6	No.7	2.4	2.6	3.6	Perfume composition of PTL3
Comp. Ex. 7	No.8	4.1	4.1	4.0	
Comp. Ex. 8	No.9	1.9	1.8	1.8	

[0043]

[Table 5]

Table 5 Evaluation comments for fragrances and flavors of perfume compositions of Example 1 and Comparative Examples 1-8 (Nos. 1-9)

5

	Perfume composition	Comment
Example 1	No. 1	Fresh, strong lemon flavor, natural quality aroma
Comp. Ex. 1	No. 2	Somewhat fresh but weak lemon flavor, and heavy different-quality and lacking natural quality, compared to perfume composition No. 1
Comp. Ex. 2	No. 3	Chlorine-like, fatty, different-quality aroma
Comp. Ex. 3	No. 4	Fresh lemon flavor, but spicy different-quality and lacking natural quality, compared to perfume composition No. 1
Comp. Ex. 4	No. 5	Strong metallic flavor, different-aroma
Comp. Ex. 5	No. 6	Somewhat fresh, but heavy different-quality and lacking natural quality, compared to perfume composition No. 1
Comp. Ex. 6	No. 7	Fresh, but lacking lemon flavor and natural quality, compared to perfume composition No. 1
Comp. Ex. 7	No. 8	Fresh, strong lemon flavor, and aroma with natural quality
Comp. Ex. 8	No. 9	Weak freshness, weak lemon flavor

[0044]

<Results>

Perfume composition No. 1 of Example 1 according to the invention and perfume composition No. 8 of Comparative Example 7 had strong lemon aromas, freshness and very natural aromatic qualities.

On the other hand, with the perfume compositions of Comparative Examples 1 to 6 and 8 (No. 2 to No. 7 and No. 9), which had blending ratios of 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one that were outside of the ranges of 0.02 to 0.4 for 4,8-dimethyl-3,7-nonadien-2-yl acetate and 0.04 to 0.8 for 4,8-dimethyl-3,7-nonadien-2-one with respect to 1 for 4,8-dimethyl-3,7-nonadien-2-ol, all received evaluation results indicating chlorine-like aromas or metallic or spicy qualities, as different aromas compared to Example 1, and lacking a lemon aroma and natural quality.

[0045]

[Example 2]

(Lemon beverage (pH = 3))

Lemon beverage 1 (pH = 3) according to the invention was obtained by a common method using the following recipe.

[Table 6]

Table 6 Lemon beverage 1 (pH = 3)

Component name	Parts by weight
	Example 2
Lemon beverage (pH = 3)	1

High-fructose corn syrup	107
Citric acid	1
Sodium citrate (adjusted to pH = 3)	q.s.
Perfume composition No. 1	1
Ion-exchanged water	remainder
Total	1000

[0046]

[Comparative Examples 9 to 11]

Perfume compositions No. 6 to 8 were added
 5 instead of perfume composition No. 1 of Example 2, to
 obtain lemon beverages (pH = 3) 2 to 4.

[0047]

[Test Example 1]

The obtained lemon beverages (pH = 3) 1 to 4
 10 were each filled into a 350 milliliter transparent PET
 bottle container and sterilized at 70°C for 10 minutes,
 and then stored for 10 days at 50°C. The fragrance and
 flavor of the stored lemon beverages (pH = 3) were
 evaluated by a panel of 10 experts based on the
 15 following evaluation criteria, and the average values
 for the results are shown in Table 7, with the comments
 obtained during the evaluation being shown in Table 8.

[0048]

Fragrance and flavor evaluation criteria:

20 Very strongly noticeable: 5

Strongly noticeable: 4

Noticeable: 3

Slightly noticeable: 2

Very weakly noticeable: 1

[0049]

[Table 7]

5

Table 7 Average values of evaluation results for
 fragrances and flavors of stored lemon beverages (pH =
 10 3)

Example	Lemon flavor	Natural quality	Freshness	Deterioration odor
Lemon beverage 1	3.3	3.3	2.4	1.2
Lemon beverage 2	2.4	2.1	1.9	1.7
Lemon beverage 3	2.6	2.9	1.9	1.7
Lemon beverage 4	1.2	1.2	1.7	2.9

[0050]

[Table 8]

15 Table 8 Evaluation comments for fragrances and flavors
 of stored lemon beverages (pH = 3)

Example	Comment
Lemon beverage 1	Lemon flavor, natural quality, low off-odor
Lemon beverage 2	Some lemon flavor, but lacking natural quality, some off-odor noticeable
Lemon beverage 3	Some lemon flavor, natural quality, but some off-odor noticeable
Lemon beverage 4	Off-odor, lacking lemon flavor, or natural quality and freshness

20 [0051]

<Results>

Lemon beverage 1 of the invention had a fresh lemon aroma with a natural quality, even after storage at 50°C for 10 days, and the distinctness of off-odor was reduced compared to lemon beverages 2 to 4.

5 [0052]

[Example 3] (Grapefruit flavor)

Perfume composition No. 10 according to the invention having the following recipe was obtained by a common method.

10 [Table 9]

Table 9 Perfume composition No.10

Component name	Parts by weight
	Example 3
Perfume composition	No.10
Grapefruit oil	900
Octanal	0.5
Decanal	0.5
Dodecanal	0.5
Citronellal	0.5
4,8-Dimethyl-3,7-nonadien-2-ol	0.55
4,8-Dimethyl-3,7-nonadien-2-yl acetate	0.05
4,8-Dimethyl-3,7-nonadien-2-one	0.1
Ethyl alcohol	remainder
Total	1000

[0053]

[Comparative Example 12]

15 Ethyl alcohol was added instead of the 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one of Example 3, to obtain perfume composition No. 11.

[0054]

20 [Example 4, Comparative Example 13]

A common method was used to obtain grapefruit beverage 1 of the invention and grapefruit beverage 2 as a comparative control, with the following recipes.

[Table 10]

5

Table 10 Grapefruit beverages (1, 2)

Component name	Parts by weight	
	Example 4	Comparative Example 13
Grapefruit beverage	1	2
1/6 Grapefruit cloudy juice	200	200
Perfume composition No. 10	1	-
Perfume composition No. 11	-	1
Ion-exchanged water	remainder	remainder
Total	1000	1000

10 [0055]

[Test Example 2]

Grapefruit beverage 1 of the invention and the comparative control 2 were comparatively evaluated in terms of fragrance and flavor, by a panel of 10 experts.

15 The average evaluation results for fragrance and flavor are shown in Table 11.

[Table 11]

Table 11 Evaluation results for fragrances and flavors of grapefruit beverages 1 and 2

Example	Fragrance/flavor evaluation
Grapefruit beverage 1	Increased natural quality and freshness compared to

	control
Grapefruit beverage 2	Control

[0056]

[Example 5] (Orange flavor)

Perfume composition No. 12 according to the
 5 invention having the following recipe was obtained by a
 common method.

[Table 12]

Table 12 Perfume composition No.12

Component name	Parts by weight
	Example 5
Perfume composition	No.12
Orange oil	700
Petitgrain oil	50
Neroli oil	50
Citraldimethylacetal	20
Valencene	5
4,8-Dimethyl-3,7-nonadien-2-ol	0.55
4,8-Dimethyl-3,7-nonadien-2-yl acetate	0.05
4,8-Dimethyl-3,7-nonadien-2-one	0.1
Ethyl alcohol	remainder
Total	1000

10

[0057]

[Comparative Example 14]

Ethyl alcohol was added instead of the 4,8-
 dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-
 15 yl acetate and 4,8-dimethyl-3,7-nonadien-2-one of
 Example 5, to obtain perfume composition No. 13.

[0058]

[Example 6, Comparative Example 15]

A common method was used to obtain orange

beverage 1 of the invention and orange beverage 2 as a comparative control, with the following recipes.

[0059]

[Table 13]

5

Table 13 Orange beverages (1, 2)

Component name	Parts by weight	
	Example 6	Comparative Example 15
Orange beverage	1	2
1/6 Orange cloudy juice	200	200
Perfume composition No. 12	1	–
Perfume composition No. 13	–	1
Ion-exchanged water	remainder	remainder
Total	1000	1000

10 [0060]

[Test Example 3]

Orange beverage 1 of the invention and the comparative control 2 were comparatively evaluated in terms of fragrance and flavor, by a panel of 10 experts.

15 The average evaluation results for fragrance and flavor are shown in Table 14.

[Table 14]

Table 14 Evaluation results for fragrances and flavors of orange beverages 1 and 2

Example	Fragrance/flavor evaluation
Orange beverage 1	Increased natural quality and top volume quality compared to control
Orange beverage 2	Control

20

[0061]

[Example 7]

Perfume composition No. 14 according to the invention having the following recipe was obtained by a common method.

5 [Table 15]

Table 15 Perfume composition No.14

10

Component name	Parts by weight
	Example 7
Perfume composition	No.14
α -Pinene	20
β -Pinene	80
γ -Terpinene	70
Linalool	35
Octanal	5
Decanal	10
Geranyl acetate	10
Methyl dihydrojasmonate	100
Galaxolide	50
4,8-Dimethyl-3,7-nonadien-2-ol	2.75
4,8-Dimethyl-3,7-nonadien-2-yl acetate	0.25
4,8-Dimethyl-3,7-nonadien-2-one	0.4
Limonene	remainder
Total	1000

[0062]

[Comparative Example 16]

15 Perfume composition No. 15 was prepared with the same recipe and method, except that limonene was added instead of 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one in Example 7.

[0063]

[Example 8, Comparative Example 17]

A common method was used to obtain shampoo 1 of the invention and shampoo 2 as a comparative control, with the following recipes.

[Table 16]

Table 16 Evaluation results for fragrances of shampoos 1 and 2

Component name	Parts by weight	
	Example 8	Comp. Ex. 17
Shampoo	1	2
Polyoxyethylene (2) lauryl ether	90	90
Sodium sulfate	40	40
Sodium lauryl sulfate	30	30
Coconut oil fatty acid amide propyl betaine	30	30
High-polymerized methylpolysiloxane	20	20
Methylpolysiloxane	10	10
Coconut oil fatty acid monoethanol amide	10	10
Propylene glycol	20	20
Ethylene glycol distearate	20	20
preservative	1	1
Perfume composition No.14	1	–
Perfume composition No.15	–	1
Purified water	remainder	remainder
Total	1000	1000

[0064]

[Test Example 4]

Shampoo 1 of the invention and shampoo 2 as the comparative control were comparatively evaluated in terms of fragrance, by a panel of 10 experts. The

average evaluation results for fragrance are shown in Table 17.

[0065]

[Table 17]

5

10 Table 17 Evaluation results for fragrances of shampoos 1 and 2

Example	Fragrance evaluation
Shampoo 1	Increased natural quality, freshness and strength of aroma, compared to control
Shampoo 2	Control

Industrial Applicability

[0066] By adding the three compounds 4,8-dimethyl-

15 3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one to perfume compositions in specific proportions according to the invention, it is possible to produce perfume compositions, fruit drinks or foods and beverages, and
 20 cosmetic products such as shampoos, having lower generation of off-odor with passage of time, compared to citral-containing perfume compositions that have similar fragrance properties.

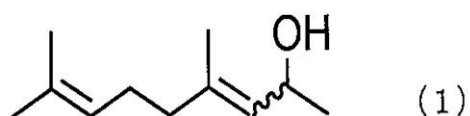
In addition, it is possible to impart a
 25 citral-like lemon aroma and freshness with a natural quality, to such products.

CLAIMS

1. A perfume composition comprising three compounds:

- 5 4,8-dimethyl-3,7-nonadien-2-ol represented by formula (1):

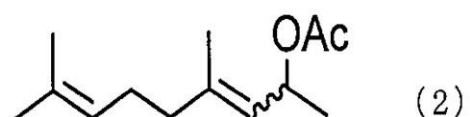
[Chemical Formula 1]



(wherein the wave line bond indicates the cis form or
10 trans form, or a mixture of the cis form and trans form in any proportion),

4,8-dimethyl-3,7-nonadien-2-yl acetate represented by formula (2):

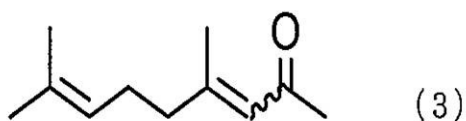
[Chemical Formula 2]



15 (wherein the wave line bond indicates the cis form or trans form, or a mixture of the cis form and trans form in any proportion, and Ac represents an acetyl group), and

- 20 4,8-dimethyl-3,7-nonadien-2-one represented by formula (3):

[Chemical Formula 3]



(wherein the wave line bond indicates the cis form or trans form, or a mixture of the cis form and trans form in any proportion),

5 combined in a proportion of 0.02 to 0.4 for 4,8-dimethyl-3,7-nonadien-2-yl acetate and 0.04 to 0.8 for 4,8-dimethyl-3,7-nonadien-2-one, with respect to 1 for 4,8-dimethyl-3,7-nonadien-2-ol.

2. A food containing a perfume composition
10 according to claim 1.

3. A beverage containing a perfume composition according to claim 1.

4. A cosmetic product containing a perfume composition according to claim 1.

15 5. A method of imparting a citral-like fragrance and flavor to a food and beverage or cosmetic product, wherein a perfume composition according to claim 1 is added to a food and beverage or cosmetic product so that the total concentration of the three
20 compounds, 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate and 4,8-dimethyl-3,7-nonadien-2-one is 0.000000000001 mass% (0.01ppt) to 0.05 mass%.

Abstract

PERFUME COMPOSITION, THE METHOD OF IMPARTING FRAGRANCE
OR FLAVOR TO FOOD, BEVERAGE, COSMETICS, AND FOOD,
BEVERAGE, COSMETICS CONTAINING SAID PERFUME COMPOSITION

5

Provided are a perfume composition, which is obtained by mixing three kinds of compounds, 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate, and 4,8-dimethyl-3,7-nonadien-2-one, which
10 are extremely effective for giving a sense of lemony citrus, a sense of natural spontaneity and a sense of fresh feeling, each in a specific proportion; and foods, beverages, perfumes and cosmetics that are formulated with the perfume composition. Provided is a perfume
15 composition that is obtained by mixing three kinds of compounds, namely 4,8-dimethyl-3,7-nonadien-2-ol, 4,8-dimethyl-3,7-nonadien-2-yl acetate, and 4,8-dimethyl-3,7-nonadien-2-one, in a specific proportion.