

COLLAGEN PEPTIDE- AND CERAMIDE-CONTAINING COMPOSITION AND METHOD OF PRODUCING THE SAME

[CROSS REFERENCE TO RELATED APPLICATIONS]

5 [0001]

This application is based on and claims the benefit of priority to Japanese patent application No. 2015-166707, filed on August 26, 2015, and the whole disclosure of which is incorporated herein by reference.

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[Technical Field]

[0002]

The present invention relates to a collagen peptide- and ceramide-containing composition, in which the occurrence of brownish discoloration during the granulation step is prevented, and a method of producing the composition.

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[Background Art]

[0003]

A collagen peptide or a ceramide is well known as an ingredient for food and drink expected to have cosmetic effects on skin (WO 2008/059927 (Patent Literature 1), WO 2010/125910 (Patent Literature 2), and JP 2008-184428 A (Patent Literature 3)). A collagen peptide used for food is obtained from a raw material such as pigskin or fish scales. In addition, a ceramide material used for food is obtained from a plant-derived raw material such as rice, wheat and Amorphophallus konjac as well as an animal-derived raw material such as egg yolk and milk.

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[0004]

Raw materials of different origins or different production methods give different compositions of the resultant materials. For example, a main ingredient of a plant-derived ceramide is a glycosyl ceramide to which glucose is bonded, while an animal-derived ceramide contains a large amount of sphingomyelins or sphingoglycolipids. In addition, even ceramide raw materials equally prepared with milk have different compositions of proteins, lipids, and minerals when they are produced via different

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purification steps.

[0005]

In recent years, there are increased needs for easy consumption of food and drink expected to have cosmetic effects on skin, and many products are on the market. The products have various forms including powder, tablet, capsule, drink, jelly drink, and gummi candy.

[0006]

Powder drink containing a collagen peptide needs to be easily soluble. As a raw material powder of a collagen peptide has poor water precipitability, a method of producing granules with instantaneous solubility has been known by separately producing granules containing a collagen peptide and granules with excellent water precipitability, and then adding an emulsifier to the granules while mixing them to obtain mixed granules coated with the emulsifier (WO 2006/117958 (Patent Literature 4)).

[Citation List]

[Patent Literatures]

[0007]

Patent Literature 1: WO 2008/059927

Patent Literature 2: WO 2010/125910

Patent Literature 3: JP 2008-184428 A

Patent Literature 4: WO 2006/117958

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[SUMMARY OF INVENTION]

[Technical Problem to be solved]

[0008]

The present inventors attempted to produce a powdery or granular composition containing a collagen peptide and a ceramide as essential components, and further containing vitamin C, a perfume or the like. As a result of search for a raw material with a high content of a sphingomyelin as the ceramide, the present inventors selected an animal-derived phospholipid material having a lipid content of 70% by weight or more in a solid content (a milk-derived phospholipid having a lipid content of 85% by weight) as a ceramide raw material.

[0009]

When a ceramide raw material having a very high lipid content is mixed directly with a powdery or granular composition, segregation occurs due to stickiness of the ceramide raw material.

5 Therefore, it is impossible to use the composition as a powder drink suitably. Then, a solution in which the ceramide raw material is dissolved in warm water is prepared and granulated while spraying onto a collagen peptide. As a result, a granular composition in which a ceramide and a collagen peptide are uniformly dispersed
10 can be obtained. However, the composition undergoes brownish discoloration during the granulation step and generates an unpleasant odor. In addition, when the composition is stored in a high-temperature zone at the latter half of 30°C or higher, the brownish discoloration proceeds and the odor is strengthened.

15 [0010]

Therefore, an object of the present invention is to provide a collagen peptide- and ceramide-containing composition, in which the collagen peptide and the ceramide are uniformly dispersed, and brownish discoloration and an unpleasant odor which will occur and
20 proceed during the granulation step and the storage in a high-temperature zone as in the summer season are prevented, as well as a method of producing the composition.

[Solution to Problem]

25 [0011]

The present inventors focused on and studied a combination of ingredients that cause brownish discoloration. A solution mixture containing a raw material other than a collagen peptide was sprayed onto a collagen peptide for fluidized bed
30 granulation. As a result, the present inventors found that an especially unpleasant odor occurs in a test section combining a collagen peptide and a ceramide raw material. Through further studies, the present inventors found that brownish discoloration and a specific odor occur even when only a not yet granulated
35 ceramide raw material is filled in an inclusion material and then stored in a high-temperature zone.

[0012]

Therefore, the present inventors further studied a method of stabilizing a ceramide raw material. As a result, the present inventors found that a complex in which the ceramide raw material is included in a cyclodextrin has very high storage stability. Furthermore, the present inventors found that by granulating while spraying the complex solution onto a collagen peptide, not only the collagen peptide and the ceramide are uniformly dispersed in the composition, but also the occurrence of both brownish discoloration and an unpleasant odor are significantly prevented at the time of the production and the storage in a high-temperature zone.

The present invention is based on the above findings.
[0013]

The present invention includes the following items.
[0014]

(1) A collagen- and ceramide-containing composition, including: a collagen peptide or collagen peptide-containing granules, and a ceramide raw material included in a cyclodextrin that is adhered onto the collagen peptide or collagen peptide-containing granules.

(2) The composition according to (1), wherein the ceramide raw material has a lipid content of 70% by weight or more in a solid content.

(3) The composition according to (1) or (2), wherein the ceramide raw material contains a milk-derived phospholipid.

(4) The composition according to any one of (1) to (3), wherein the cyclodextrin is a γ -cyclodextrin, a β -cyclodextrin, or an α -cyclodextrin.

(5) The composition according to any one of (1) to (4), wherein the cyclodextrin is a γ -cyclodextrin.

(6) The composition according to any one of (1) to (5), having a weight ratio of the ceramide raw material to the cyclodextrin of 1 : 0.5 to 1 : 5.

(7) A method of producing a collagen- and ceramide-containing composition, including the steps of: obtaining a solution mixture containing a ceramide raw material and a cyclodextrin; and granulating while spraying the solution mixture onto a collagen peptide or collagen peptide-containing granules.

Advantageous Effects of Invention

[0015]

5 The present invention can provide a collagen peptide- and ceramide-containing composition, in which the collagen peptide and the ceramide are uniformly dispersed, and the occurrence of brownish discoloration and an unpleasant odor during the granulation step are prevented, as well as a method of producing the composition. Furthermore, the composition of the present
10 invention can prevent the occurrence of brownish discoloration and an unpleasant odor during storage in a high-temperature zone as in the summer season. According to the present invention, even if the ceramide has a high lipid content of 70% by weight or more in a solid content, a composition having the above satisfactory
15 characteristics can be obtained.

[BRIEF DESCRIPTION OF DRAWINGS]

[0016]

20 Fig. 1 shows a differential scanning calorimetry (DSC) chart for samples having different weight ratios of a ceramide raw material to a cyclodextrin in Test example 4. In the spectrum, the horizontal axis indicates Temperature (°C), and the vertical axis indicates DSC (μW).

25 Fig. 2 shows an NMR spectrum for samples having different weight ratios of a ceramide raw material to a cyclodextrin in Test example 5. In the spectrum, the horizontal axis indicates chemical shift (ppm), and the vertical axis indicates relative intensity.

30 Fig. 3 shows an NMR spectrum for samples having different weight ratios of a ceramide raw material to a cyclodextrin in Test example 5.

[MODE FOR CARRYING OUT THE INVENTION]

[0017]

35 In the present invention, "a collagen peptide- and ceramide-containing composition" contains at least "a ceramide raw material", "a cyclodextrin", and "a collagen peptide" described below.

[0018]

[Ceramide raw material]

In the present invention, "ceramide" refers to a type of sphingolipid having a structure in which a sphingosine, a fatty acid, and a sugar are bonded, and is originated from a natural material. Examples of a ceramide include animal-derived materials extracted from milk, egg or the like, plant-derived materials extracted from wheat, rice, soybean, Amorphophallus konjak, corn, or the like, and microorganism-derived materials extracted from yeast or the like. The ceramide can be obtained according to known extraction methods. In addition to the known extraction methods, the ceramide can be obtained via a coating process using an emulsifier or the like. In the present invention, "a ceramide raw material" contains the ceramide.

[0019]

Preferably, the ceramide raw material in the present invention has a lipid content of 70% by weight or more in a solid content. In the present invention, a ceramide raw material having a lipid content of 80% by weight or more, further 85% by weight or more in a solid content can also be used.

[0020]

In the present invention, the ceramide raw material preferably contains "a milk-derived phospholipid". The milk-derived phospholipid refers to a product extracted from milk and contains a phosphatidylcholine, a phosphatidylethanolamine, and a sphingomyelin as main ingredients. In the present invention, the milk-derived phospholipid broadly refers to a phospholipid extracted from milk, and a commercial product is also available.

[0021]

In the present invention, a ceramide raw material preferably contains a sphingomyelin. In particular, a ceramide raw material containing 15% by weight or more of a sphingomyelin is preferred.

[0022]

[Cyclodextrin]

Examples of the cyclodextrin preferably used in the composition of the present invention include a γ -cyclodextrin, a β -

cyclodextrin, and an α -cyclodextrin. A γ -cyclodextrin or an α -cyclodextrin is more preferred, and a γ -cyclodextrin is most preferred. A cyclodextrin is a compound having a steric structure having a cyclic sugar chain, and the external surface is hydrophilic
5 and the internal surface is lipophilic.

[0023]

[Solution mixture]

In the method of producing the composition of the present invention, "a solution mixture" refers to a solution in which at least
10 the ceramide raw material and the cyclodextrin are dissolved in a solvent such as water. The solvent used in the solution mixture is preferably water. The temperature of the solvent may be, but not limited to, 40°C or higher for easy dissolution and is preferably in the range of 40°C to 80°C. The preferred amount of the solvent
15 varies according to the type of cyclodextrin. The amount of the solvent when a γ -cyclodextrin is used may be 1.5 times or more by weight, and is preferably about 2.5 to 5 times by weight and more preferably about 3 to 4 times by weight relative to the solid content of components (a cyclodextrin, a ceramide raw material, and
20 components optionally added) in the solution mixture. The amount of the solvent when an α -cyclodextrin is used may be 5 times or more by weight, and is preferably 6.5 times or more by weight relative to the solid content of components in the solution mixture. The amount of the solvent when a β -cyclodextrin is used may be
25 10 times or more by weight, and is preferably 20 times or more by weight relative to the solid content of components in the solution mixture.

[0024]

Although details of the action are unknown, it is presumed
30 that when a ceramide raw material contacts a cyclodextrin in a solution mixture and then the solution mixture is dried, the ceramide raw material is included inside of the cyclodextrin. It is presumed that a brownish discoloration reaction caused by the ceramide raw material during the granulation step is prevented by
35 formation of a inclusion complex in such a manner. Further, the presumption is on a theoretical basis, and does not restrict the present invention.

[0025]

The weight ratio of the ceramide raw material to the cyclodextrin in the solution mixture is preferably 1 : 0.5 to 1 : 5, more preferably 1 : 1 to 1 : 2, and even more preferably 1 : 1.5 to 1 : 2. A higher weight ratio of the cyclodextrin in the solution mixture tends to prevent the occurrence of brownish discoloration and odor. In addition, a higher weight ratio of the cyclodextrin in the solution mixture results in higher fluidity of the resultant composition. However, if the weight ratio of the cyclodextrin in the solution mixture is high, the amount of the solvent (for example, water) required for spraying a binder solution with appropriate viscosity is increased. When the amount of water in a solution mixture is large, heat quantity required for drying the solution mixture at granulation is increased, and therefore, the production cost is increased. Accordingly, the weight ratio of the cyclodextrin in the solution mixture is preferably within the range mentioned above.

[0026]

The solution mixture can contain other components as long as the effects of the present invention are not impaired. Examples of such other components include binding agents such as gum arabic, pullulan, guar gum, and starch; cores such as dextrin, starch, crystalline cellulose, and glucide raw materials; antioxidants including plant extracts such as catechins, vitamins, rosemary extract, and Myrica rubra extract; emulsifiers; brightening agents; and perfumes.

[0027]

[Collagen peptide]

Collagen is produced by extraction from the skin, bone, ligament, tendon, cartilage or the like produced as a byproduct in processing livestock such as cattle and pigs, or fish. In the present invention, "a collagen peptide" is obtained by decomposing the extracted collagen or gelatin through an enzyme, a chemical treatment or the like. The origin of the collagen peptide can be selected appropriately, but a fish-derived collagen peptide is preferred.

[0028]

The molecular weight of the collagen contained in the collagen peptide can be selected appropriately. A low-molecular weight collagen component may have high reactivity, but can be incorporated into the composition of the present invention without
5 occurrence of brownish discoloration during the granulation step. For example, a collagen peptide having an average molecular weight of 1800 to 5000 can be suitably used as a collagen peptide.
[0029]

[Collagen peptide-containing granules]

10 Collagen peptide-containing granules can be obtained by spraying a binder solution containing a binding agent prepared separately (preferably gum arabic) onto the collagen peptide, and granulating the resultant product. The binder solution can contain
15 other components as long as the effects of the present invention are not impaired. Examples of such other components include glucide raw materials, lactobacillus powders, amino acid powders, vitamin powders, perfumes, emulsifiers, crystalline cellulose, coloring agents, sweetening agents, acidifiers, and lubricants. Examples of the granulation method include fluidized bed
20 granulation, extruding granulation, compression granulation, and spray drying, and especially fluidized bed granulation is preferred. Collagen peptide-containing granules may be obtained by optionally performing a drying step or a cooling step after the granulation step.

25 [0030]

[Composition]

The composition of the present invention can be obtained by spraying a solution mixture containing a ceramide raw material and a cyclodextrin onto the collagen peptide or collagen peptide-
30 containing granules and then granulating the resultant product. The composition of the present invention has a configuration that a ceramide raw material included in a cyclodextrin is adhered onto a collagen peptide or collagen peptide-containing granules.

[0031]

35 That is, the composition of the present invention is in a state where a ceramide raw material included in a cyclodextrin is adhered onto a collagen peptide or collagen peptide-containing

granules, and a coating layer of the ceramide raw material included in the cyclodextrin is formed. In this state, the whole or part of the collagen peptide or the collagen peptide-containing granules is coated with the coating layer. Accordingly, it can be said that the composition of the present invention has a coating layer made from a ceramide raw material included in a cyclodextrin on the whole or part of the collagen peptide or the collagen peptide-containing granules.

[0032]

That is, as described above, the composition of the present invention can be produced by a method including the steps of: obtaining a solution mixture containing a ceramide raw material and a cyclodextrin; and granulating while spraying the solution mixture onto a collagen peptide or collagen peptide-containing granules.

[0033]

The composition of the present invention can be produced by further adding a binding agent (for example, gum arabic) to a solution mixture containing a ceramide raw material and a cyclodextrin, and granulating while spraying the solution mixture onto a collagen peptide or collagen peptide-containing granules. Examples of the granulation method include fluidized bed granulation, extruding granulation, compression granulation, and spray drying, and especially fluidized bed granulation is preferred. The composition of the present invention can also be obtained by optionally performing a drying step or a cooling step after the granulation step.

[0034]

In the composition of the present invention, the weight ratio among the collagen peptide, the ceramide raw material, and the cyclodextrin is not limited. In the composition of the present invention, the collagen peptide and the ceramide are dispersed uniformly, even if the amount of ceramide is minute relative to that of collagen peptide, the amount of collagen peptide is minute relative to that of ceramide, or the amount of collagen peptide and the amount of ceramide are nearly equal to each other.

[0035]

Other components can be added to the composition of the present invention in addition to the collagen peptide, the ceramide raw material, and the cyclodextrin as long as the effects of the present invention are not impaired. Examples of such other components include binding agents such as gum arabic, glucide raw materials, glucosamine, N-acetylglucosamine, hyaluronic acid, chondroitin sulfate, coenzyme Q10, fermented milk serum products, plant extracts, lactobacillus powders, amino acid powders, vitamin powders, sweetening agents, perfumes, emulsifiers, lubricants, and brightening agents. The components can be added before, during, or after the granulation step of granulating while spraying the solution mixture containing the ceramide raw material and the cyclodextrin onto the collagen peptide or collagen peptide-containing granules for granulation.

[0036]

The composition of the present invention can be obtained via a tableting step or a compression step in addition to the mixing step of granules. The tableting step and the compression step can be performed by a known means. Properties of "the composition" of the present invention may be mixed granules, mixed granules containing additional powder components or the like, a tablet obtained by tableting the granules or the like, or a compression molded body obtained by compression-molding the granules or the like.

[0037]

The composition of the present invention can be used as it is as powder drink, tablet, or the like, because the occurrence of brownish discoloration and an odor is prevented during the granulation step and the storage in a high-temperature zone. The composition of the present invention can also be used as a raw material for any other forms of products such as gummi candy, jelly drink, and packaged drink. The composition of the present invention has good fluidity and is less prone to powder scattering in production of any other forms of products, and thus the composition is excellent in handleability in production.

[EXAMPLES]

[0038]

The present invention is described in detail by the following examples, however, these examples should not be understood to limit the present invention.

5 [0039]

Comparative example 1: A composition produced by granulating while spraying directly a ceramide raw material onto a collagen peptide

[Base powder]

10 Collagen peptide (a collagen peptide manufactured by Nitta Gelatin Inc., molecular weight: about 3000): 150 parts by weight

Sodium ascorbate: 10 parts by weight

Trehalose fine powder: 100 parts by weight

Maltodextrin: 40 parts by weight

15 [0040]

[Binder solution]

To 40 parts by weight of stirred water, 0.5 parts by weight of gum arabic and 10 parts by weight of a ceramide raw material (a milk-derived phospholipid having a lipid content of 85% by weight, manufactured by Fonterra (Japan) Limited) were added and dissolved.

[0041]

25 While the base powder was charged into a fluidized bed granulating machine and fluidized at 85°C, the binder solution was sprayed onto the base powder to perform granulation. After the granules obtained by granulation were dried at 100°C for 30 minutes, 80 parts by weight of a maltodextrin were added and mixed, and 2.5 parts by weight of a perfume and 2.5 parts by weight of an emulsifier were sprayed onto the mixture to obtain the composition of Comparative example 1.

30 The resultant composition was granular, and a ceramide and a collagen peptide were uniformly dispersed in the composition. [0042]

35 In the composition of Comparative example 1, brownish discoloration and an unpleasant odor occurred during granulation in the fluidized bed granulating machine. Furthermore, when the resultant composition was enclosed in an aluminum pouch and

stored at 23°C, 37°C, and 60°C for a week, the brownish discoloration proceeded and the unpleasant odor was strengthened in all of the storage temperature zones.

[0043]

5 Test example 1: Study of a combination of ingredients that cause brownish discoloration

Samples were prepared for each test section as shown in Table 1. Raw materials used in this test example were the same as those used in Comparative example 1. In a test section 1-1, an
10 untreated collagen peptide was used. A sample of a test section 1-2 was prepared by drying 400 parts by weight of a collagen peptide at 100°C for 30 minutes. Samples of test sections 1-3 to 1-8 were prepared by charging a base powder raw material into a fluidized bed granulating machine, spraying a binder solution to the raw
15 material, granulating the raw material while fluidizing the raw material at 85°C for granulation, and then drying the resultant granules at 100°C for 30 minutes.

[0044]

Each of the resultant samples was evaluated for the odor and color tone immediately after production. Furthermore, each
20 aqueous solution obtained by dissolving about 1.2 g of each sample in 150 mL of water was evaluated for the flavor and color tone.

Table 1 shows the results.

[0045]

25 [Table 1]

Test section (numbers are in parts by weight)		1-1	1-2	1-3	1-4	1-5	1-6	1-7	1-8
Base powder	Collagen peptide	400	400	400	400	400	400	–	380
	Maltodextrin	–	–	–	–	–	–	370	–
	Sodium ascorbate	–	–	–	–	–	–	–	20
Binder solution	Water	–	–	40	40	40	40	130	100
	Gum arabic	–	–	–	1.6	–	1.6	–	1.5
	Ceramide raw material	–	–	–	–	22	22	28	–
Sample (powder or granules)	Odor	Odorless	Odorless	Odorless	Odorless	<u>Strong unpleasant odor</u>	<u>Strong unpleasant odor</u>	Odorless	Odorless
	Color tone	White	White	White	White	Yellow	Yellow	Yellow (lighter than in 1-5)	White
Aqueous solution of the above samples (drink)	Flavor	Tasteless	Almost same as in 1-1	Almost same as in 1-1	Almost same as in 1-1	<u>Unpleasant flavor and odor</u>	<u>Unpleasant flavor and odor</u>	Nearly tasteless	Almost same as in 1-1
	Color tone	Transparent	Transparent	Transparent	Transparent	Cloudy (no remarkable difference among 1-5 to 1-7)	Cloudy (no remarkable difference among 1-5 to 1-7)	Cloudy (no remarkable difference among 1-5 to 1-7)	Transparent

[0046]

Table 1 shows that granules in which a collagen peptide and a ceramide raw material were bonded under a condition where moisture existed (test sections 1-5 and 1-6) had an unpleasant odor and coloration. An aqueous solution (drink) prepared by dissolving each of the samples of the test sections 1-5 and 1-6 had an unpleasant flavor and odor.

[0047]

10 Test example 2: Compositions containing collagen peptides with different molecular weights

Compositions were prepared with collagen peptides each having an average molecular weight of about 5,000, about 3,000, about 2,000, or about 1,800 (all manufactured by Nitta Gelatin Inc.) as a raw material. While the base powder (280 parts by weight of the collagen peptide) was charged into a fluidized bed granulating machine and fluidized, the binder solution (30 parts by weight of water and 17 parts by weight of the same ceramide raw material as that of Comparative example 1) was sprayed onto the base powder to perform granulation at 85°C, and then the mixture was dried at 100°C for 30 minutes. Each of the resultant compositions was enclosed in an aluminum pouch and stored at 23°C, 40°C, and 60°C for a week. Each composition after storage was evaluated for the odor and color difference.

25 Table 2 shows the evaluation results of color difference.

[0048]

[Table 2]

Molecular weight of collagen peptide	Storage temperature	Lab value (average of three measurements)			Color difference from 23°C for 1 W		
		L	a	b	ΔL	Δa	Δb
Average molecular weight about 5000	23°C1W	96.087	-0.667	13.863	0.000	0.000	0.000
	40°C1W	95.953	-0.633	14.407	-0.133	0.033	0.543
	60°C1W	92.180	2.127	19.550	-3.907	2.793	5.687
Average molecular weight about 3000	23°C1W	94.613	-0.570	16.923	0.000	0.000	0.000
	40°C1W	94.440	-0.480	17.587	-0.173	0.090	0.663
	60°C1W	90.663	2.220	23.750	-3.950	2.790	6.827
Average molecular weight about 2000	23°C1W	96.550	-0.900	14.133	0.000	0.000	0.000
	40°C1W	96.427	-0.820	14.483	-0.123	0.080	0.350
	60°C1W	93.000	1.700	19.490	-3.550	2.600	5.357
Average molecular weight about 1800	23°C1W	94.087	-0.750	16.130	0.000	0.000	0.000
	40°C1W	94.003	-0.733	16.537	-0.083	0.017	0.407
	60°C1W	91.440	1.160	21.333	-2.647	1.910	5.203

[0049]

The results show a tendency that when granules formed by spraying a ceramide raw material dissolved in water onto a collagen peptide were stored in a high-temperature zone at 40°C or higher, brownish discoloration proceeded. The brownish discoloration proceeded irrespective of the different average molecular weights of the collagen peptides. Furthermore, all of the samples after storage had an unpleasant odor like fish bait.

[0050]

10 Test example 3: Storage test of a ceramide raw material

A ceramide raw material (a milk-derived phospholipid having a lipid content of 85% by weight, manufactured by Fonterra (Japan) Limited) was enclosed in an aluminum pouch, and stored at 40°C for a week, and at 60°C for 4 days. Each of the ceramide raw materials after storage had brownish discoloration and an unpleasant odor like fish bait.

[0051]

Example 1

[Base powder]

20 Maltodextrin: 300 parts by weight

Collagen peptide (a collagen peptide manufactured by Nitta Gelatin Inc., molecular weight: about 3000): 370 parts by weight

Glucide raw material (trehalose fine powder): 175 parts by weight

25 [Binder solution]

To 100 parts by weight of stirred water, 2.0 parts by weight of gum arabic was added and dissolved.

[0052]

[Solution mixture]

30 A solution mixture was prepared by mixing components shown in Table 3. The details of the components in Table 3 are as follows.

Ceramide raw material: a milk-derived phospholipid having a lipid content of 85% by weight, manufactured by Fonterra (Japan) Limited

35 γ -cyclodextrin: trade name "CAVAMAX(R) W8 Food", manufactured by Cyclochem co., Ltd.

β -cyclodextrin: trade name "CAVAMAX(R) W7 Food",
manufactured by Cyclochem co., ltd.

α -cyclodextrin: trade name "CAVAMAX(R) W6 Food",
manufactured by Cyclochem co., ltd.

5 [0053]

[Table 3]

Components in solution mixture (parts by weight)	Example 1-1	Example 1-2	Example 1-3	Example 1-4	Example 1-5
Ceramide raw material	22	22	22	22	22
γ -Cyclodextrin	44	33	22	—	—
β -Cyclodextrin	—	—	—	44	—
α -Cyclodextrin	—	—	—	—	44
Water (70°C)	200	200	200	1500	400

[0054]

10 While the base powder was fluidized in a fluidized bed
granulating machine at 85°C, a binder solution was sprayed onto
the base powder. Next, each of the solution mixtures of Examples
1-1 to 1-5 shown in Table 3 was sprayed and dried at about 100°C
for about 10 minutes, and each of the resultant products was
15 cooled to obtain compositions 1-1 to 1-5. It took about 20 minutes
in Examples 1-1 to 1-3, about 180 minutes in Example 1-4, and
about 70 minutes in Example 1-5 to finish spraying.

[0055]

20 To 94.5 parts by weight of each of the resultant
compositions 1-1 to 1-3, an additional powdery component
(sodium ascorbate: 2.4 parts by weight) was mixed. Next, 1.0 part
by weight of a perfume and 0.6 parts by weight of an emulsifier
were sprayed onto the mixture to obtain compositions 1-1a to 1-3a.

[0056]

25 Furthermore, to 67 parts by weight of each of the
compositions 1-1 to 1-3, the following additional powdery
components were mixed.

Maltodextrin: 30 parts by weight

Sodium ascorbate: 1.5 parts by weight

Next, 0.8 parts by weight of a perfume and 0.6 parts by weight of an emulsifier were sprayed onto the mixture to obtain compositions 1-1b to 1-3b.

[0057]

In all of the compositions, a ceramide and a collagen peptide were uniformly dispersed. Additionally, all of the compositions were produced without occurrence of brownish discoloration and an unpleasant odor during the production process.

[0058]

About 3.0 g of each of the compositions immediately after production was weighed and dissolved in 150 mL of water to prepare a drink.

A composition (1-5) produced using an α -cyclodextrin was a little insoluble, and a composition (1-4) produced using a β -cyclodextrin was insoluble. On the other hand, all of the compositions (1-1 to 1-3, 1-1a to 1-3a, and 1-1b to 1-3b) produced using a γ -cyclodextrin had good dispersibility and precipitability. All of the drinks prepared with the compositions had good quality with a cloudy color and a slightly milk-like flavor.

[0059]

Additionally, each of the compositions 1-1a to 1-3a and the compositions 1-1b to 1-3b was enclosed in an aluminum pouch and stored at 60°C for 4 days and at 40°C or 23°C for 2 months. Similarly, the compositions 1-4 to 1-5 were also stored at 60°C for 4 days and at 40°C or 23°C for 2 weeks. In all of the compositions after storage, neither brownish discoloration nor an unpleasant odor occurred.

[0060]

Test example 4: Analysis (1) DSC of inclusion action of a cyclodextrin to include a ceramide raw material

A thermal behavior of samples obtained by changing the weight ratio of a ceramide raw material to a cyclodextrin was evaluated using a differential scanning calorimetry (DSC).

[0061]

Samples for measurement having weight ratios as shown in

the following table were prepared according to the following procedures, using a ceramide raw material (a milk-derived phospholipid having a lipid content of 85% by weight, manufactured by Fonterra (Japan) Limited) and a γ -cyclodextrin (γ -CD; trade name: CAVAMAX(R) W8 Food, manufactured by Cyclochem co., ltd.).

[0062]

[Table 4]

Sample No.	Weight ratio (ceramide raw material : γ -cyclodextrin)
Sample(1)	1:0 (Ceramide raw material alone)
Sample(2)	2:1
Sample(3)	1.5:1
Sample(4)	1.25:1
Sample(5)	1.0625:1
Sample(6)	1.03125:1
Sample(7)	1:1
Sample(8)	1:1.5
Sample(9)	1:2
Sample(10)	0:1 (γ -Cyclodextrin alone)

10 [0063]

As for samples (1) and (10), the raw material was subjected to the measurement as it is.

As for samples (2) to (9), the raw materials were weighted so that they could have a specific weight ratio, added to warm water at about 70°C in an amount of 4 times by weight the solid content, and stirred with a stirrer for 10 minutes, and then the resultant mixture solution was spray dried at a blast temperature of 100°C in a fluidized bed granulating machine to obtain each sample.

[0064]

20 EXSTAR6000 manufactured by Seiko Instruments Inc. was used as a DSC measurement device. The measured weight was 5.0 mg, the measuring environment was air, and the temperature rise rate was 2°C/min.

[0065]

25 The analysis results are as shown in Fig. 1.

[0066]

As shown in Fig. 1, an endothermic peak around 150°C of sample (1), a ceramide raw material alone, shifted to a high

temperature side as the weight ratio of a cyclodextrin in the sample increased, and disappeared around about 170°C (sample (6) 1.03125 : 1 to sample (7) 1 : 1). In sample (8) and sample (9), no endothermic peak was observed.

5 [0067]

The results show that the inclusion action of a cyclodextrin (γ -CD) to include a ceramide raw material (a milk-derived phospholipid having a lipid content of 85% by weight) was found in a range in which the weight ratio of a ceramide raw material to a cyclodextrin was at least 2 : 1 (1 : 0.5) to 1 : 2. Additionally, it was considered that all of the ceramide raw material used in the samples was included in the cyclodextrin around a weight ratio of a ceramide raw material to a cyclodextrin of about 1 : 1. Additionally, it was considered that all of the ceramide raw material used in the samples was included in the cyclodextrin in a range in which the weight ratio of a ceramide raw material to a cyclodextrin was 1.03125 : 1 to 1 : 2.

[0068]

Accordingly, it was verified that the inclusion action exhibited the effects of preventing brownish discoloration and occurrence of an unpleasant odor in the composition of the present invention as shown in Example 1 (a ceramide raw material : a cyclodextrin = 1 : 1 to 1 : 2 in the composition).

[0069]

25 Test example 5: Analysis (2) NMR of inclusion action of a cyclodextrin to include a ceramide raw material

A solution mixture and a dry powder thereof prepared by changing the weight ratio of a ceramide raw material to a cyclodextrin were evaluated by nuclear magnetic resonance spectrometry.

30 [0070]

Each ceramide raw material (a milk-derived phospholipid having a lipid content of 85% by weight, manufactured by Fonterra (Japan) Limited) and a cyclodextrin (γ -CD; trade name "CAVAMAX(R) W8 Food", manufactured by Cyclochem co., Ltd.) were mixed in heavy water in a weight ratio of 10 : 1, 5 : 1, 2 : 1, 1 : 1, 0.8 : 1, 0.6 : 1, 0.4 : 1, 0.2 : 1, or 0 : 1 (γ -CD alone),

heated to 70°C, and stirred to obtain a solution mixture. Then, each solution mixture was allowed to cool to room temperature, and used as a measurement sample.

[0071]

5 Additionally, a ceramide raw material (the same as the above) and a cyclodextrin (the same as the above) were mixed in water in a weight ratio of 1 : 2 and 0 : 1 (γ -CD alone) and heated to 70°C, and each of the resultant solution mixture was lyophilized and then crushed to obtain a dry powder. Each of the resultant dry
10 powders was dissolved in heavy water to perform nuclear magnetic resonance spectrometry, and used as a measurement sample similarly as above.

[0072]

 The resultant measurement sample was subjected to ^1H -
15 NMR nuclear magnetic resonance spectrometry.

 An Avance500 nuclear magnetic resonance spectrometry device manufactured by Bruker BioSpin K.K. (a ^1H resonance frequency of 500 MHz) was used for analysis and the measurement was performed at 25°C.

20 [0073]

 Fig. 2 shows the analysis results obtained using the solution mixture as a measurement sample.

[0074]

 According to the prior study, it was expected that when a
25 milk-derived phospholipid is included in a γ -cyclodextrin, an interaction occurs between H3 and H5 existing within the γ -cyclodextrin, and a chemical shift of the signals is changed. It was expected that the higher the ratio of the milk-derived phospholipid to the γ -cyclodextrin is, the greater the change of the chemical
30 shift is.

[0075]

 However, in Fig. 2, the amount of chemical shift of H3 and H5 is almost the same among the samples having different ratios of the milk-derived phospholipid to the γ -cyclodextrin. Therefore,
35 it was considered that in the measured solution mixture, the milk-derived phospholipid was not included in the γ -cyclodextrin.

 That is, it was suggested that in the composition of the

present invention containing collagen and a ceramide, no inclusion complex was formed in the production process for obtaining the solution mixture containing a ceramide raw material (a milk-derived phospholipid) and a cyclodextrin (a γ -cyclodextrin).

5 [0076]

Fig. 3 shows analysis results obtained using the solution in which a dry powder is dissolved as a measurement sample.

[0077]

The results suggested that no inclusion complex was
10 formed in the solution even when the inclusion complex, which was obtained by the step of lyophilizing the solution mixture containing a milk-derived phospholipid and a γ -cyclodextrin, and then grinding the mixture, was subjected to the analysis.

[0078]

15 Additionally, a solution mixture containing an α -cyclodextrin and a milk-derived phospholipid and a solution mixture containing a β -cyclodextrin and a milk-derived phospholipid were also prepared, and actually confirmed for the inclusion capacity in a solution containing a milk-derived phospholipid and a cyclodextrin.
20 As a result, it was found that the inclusion capacity in these solution mixtures is very low. Therefore, it was considered that the presence or absence of inclusion complex formation in both the solution containing an α -cyclodextrin and the solution containing a β -cyclodextrin is similar to that in the solution containing a γ -
25 cyclodextrin.

[0079]

Accordingly, from the results as shown in Fig. 2 and Fig. 3, it was considered that a inclusion complex is not formed merely by contact of a ceramide raw material with a cyclodextrin in a solution
30 mixture, and the ceramide raw material comes to be included within the cyclodextrin only after the solution mixture is dried.

CLAIMS

1. A collagen- and ceramide-containing composition, comprising: a collagen peptide or collagen peptide-containing granules, and a ceramide raw material included in a cyclodextrin that is adhered onto the collagen peptide or collagen peptide-containing granules.
5
2. The composition according to claim 1, wherein the ceramide raw material has a lipid content of 70% by weight or more in a solid content.
10
3. The composition according to claim 1 or 2, wherein the ceramide raw material contains a milk-derived phospholipid.
15
4. The composition according to any one of claims 1 to 3, wherein the cyclodextrin is a γ -cyclodextrin, a β -cyclodextrin, or an α -cyclodextrin.
- 20 5. The composition according to any one of claims 1 to 4, wherein the cyclodextrin is a γ -cyclodextrin.
6. The composition according to any one of claims 1 to 5, having a weight ratio of the ceramide raw material to the cyclodextrin of 1 : 0.5 to 1 : 5.
25
7. A method of producing a collagen- and ceramide-containing composition, comprising the steps of: obtaining a solution mixture containing a ceramide raw material and a cyclodextrin; and granulating while spraying the solution mixture onto a collagen peptide or collagen peptide-containing granules.
30