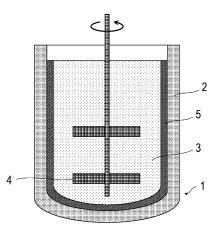
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(54) TOBACCO-COMPONENT-CONCENTRATED LIQUID, METHOD FOR MANUFACTURING SAME, FLAVOR-PRODUCING ARTICLE, AND METHOD FOR MANUFACTURING SAME

(57) Provided is a method for manufacturing an inexpensive tobacco-component-concentrated liquid, the method making it possible to concentrate a flavor component to a high degree of concentration while adequately retaining the flavor component. This method for manufacturing a tobacco-component-concentrated liquid includes a step for concentrating a tobacco-component-containing liquid through a progressive freeze concentration method.

Fig. 1



Processed by Luminess, 75001 PARIS (FR)

Description

TECHNICAL FIELD

⁵ **[0001]** The present invention relates to a tobacco-component-concentrated liquid and a method for manufacturing the tobacco-component-concentrated liquid, as well as a flavor-producing article and a method for manufacturing the flavor-producing article.

BACKGROUND ART

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[0002] Extraction of a tobacco component from a tobacco raw material is performed to improve the flavor of the tobacco raw material and decrease the amount of another component in the tobacco raw material.

[0003] For example, Patent Literature 1 discloses a method for manufacturing a tobacco raw material with a good flavor by performing extraction from a leaf tobacco material with a low-polarity solvent and further extraction from the residue with a high-polarity solvent and adding back the extract extracted with the low-polarity solvent to the residue. Patent Literature 2 discloses a method for manufacturing a tobacco product containing a lower amount of phenolic

- compound by performing extraction from a tobacco material with a solvent to provide an extract and a residue, treating the extract with phenol oxidase to decrease the amount of phenolic compound, and then combining the extract with the residue. Patent Literature 3 discloses a method of mixing, as an essential oil, a fraction produced by steam distillation of leaf tobacco with another material. Patent Literature 4 discloses a method for preparing a distillate by vacuum distillation
- of leaf tobacco with another material. Patent Literature 4 discloses a method for preparing a distillate by vacuum distillation of a tobacco raw material.

CITATION LIST

25 PATENT LITERATURE

[0004]

- PTL 1: International Publication No. 2015/029977
- PTL 2: Japanese Unexamined Patent Application Publication (Translation of PCT Application) No. 2002-520005
- PTL 3: Japanese Examined Patent Application Publication No. 60-045909

PTL 4: Chinese Patent Application Publication No. 104757703

SUMMARY OF INVENTION

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TECHNICAL PROBLEM

[0005] An extract, a fraction, or a distillate manufactured by such a method contains an extracting solvent, such as water, and, when added to a tobacco product as it is, it is necessary to remove the extracting solvent by vaporization. However, such a removal operation is energy expensive. Thus, an extract or the like may be concentrated in advance before being added to a tobacco product. This concentration operation not only obviates the need for the removal operation but also decreases the volume of the extract or the like, thus reducing the transportation cost and the storage

- cost. Furthermore, when the extracting solvent is water, the concentration operation can decrease the water content, prevent or suppress the growth of microorganisms in the liquid, and improve the storage stability.
- ⁴⁵ **[0006]** A generally and widely used concentration method for a solution is, for example, an evaporative concentration method, a membrane concentration method, or a freeze-drying method. The evaporative concentration method is a method for heating a solution to vaporize and remove a solvent from the solution. This method can be performed with a simple apparatus and can reduce the apparatus cost. When the method is used to concentrate an extract or the like containing a tobacco component, however, heating may volatilize and disperse or alter the quality of a component useful
- for imparting a flavor (hereinafter referred to as a flavor component) contained in the tobacco component. Furthermore, removal of the solvent requires a lot of energy.
 [0007] The membrane concentration method is a method for applying pressure to a solution using a reverse osmosis membrane (RO membrane) or the like to separate a solvent from the solution. This method can be performed at normal temperature, can prevent or suppress thermal alteration of a component in a solution, causes no phase change in the concentration and separation process, and can therefore consume less energy. However, concentration at a high con-
- ⁵⁵ concentration and separation process, and can therefore consume less energy. However, concentration at a high concentration is difficult, and the concentration operation at normal temperature may cause microbial contamination. Furthermore, cleaning and replacing the membrane take time and cost.

[0008] The freeze-drying method is a method for sublimating a solvent from a frozen raw material to remove the

solvent. This method is also often used to concentrate a liquid. In the method, the solvent in the raw material is solid throughout the entire drying period and does not move in liquid form in the raw material. Furthermore, drying at a low temperature can prevent or suppress thermal alteration and chemical change of a material. However, the method still has many problems to be technically overcome, is expensive in terms of energy consumption and capital investment, and is therefore practically applied only to some foods, such as fruits, at present.

5 [0009] It is an object of the present invention to provide an inexpensive method for manufacturing a tobacco-componentconcentrated liquid, the method making concentration possible while adequately retaining the flavor component, and a tobacco-component-concentrated liquid manufactured by the method, as well as a method for manufacturing a flavorproducing article including the method for manufacturing a tobacco-component-concentrated liquid, and a flavor-pro-10

ducing article manufactured by the method.

SOLUTION TO PROBLEM

- **[0010]** The present invention includes the following aspects.

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[1] A method for manufacturing a tobacco-component-concentrated liquid, comprising the step of concentrating a liquid containing a tobacco component by a progressive freeze concentration method.

[2] The method according to [1], further comprising the step of extracting the tobacco component in a tobacco raw material with a solvent to manufacture the liquid containing the tobacco component before the step of concentrating the liquid containing the tobacco component by the progressive freeze concentration method.

[3] The method according to [2], wherein the solvent contains water.

[4] The method according to any one of [1] to [3], further comprising the step of filtering the liquid containing the tobacco component to remove a solid before the step of concentrating the liquid containing the tobacco component by the progressive freeze concentration method.

- 25 [5] The method according to any one of [1] to [4], wherein a total area of peaks of a compound group with an RI of 2000 or less is 70% or more of a total area of all peaks in a chromatogram obtained by gas chromatography-mass spectrometry (GC/MS) when the tobacco-component-concentrated liquid is subjected to headspace analysis. [6] A tobacco-component-concentrated liquid manufactured by the method according to any one of [1] to [5].
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manufacturing a tobacco-component-concentrated liquid by the method according to any one of [1] to [5]; applying the tobacco-component-concentrated liquid to a substrate and drying the tobacco-component-concentrated liquid to manufacture a tobacco-component-containing substrate; and

manufacturing a flavor-producing article including the tobacco-component-containing substrate.

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[8] A flavor-producing article manufactured by the method according to [7].

[7] A method for manufacturing a flavor-producing article, comprising the steps of:

[9] The flavor-producing article according to [8], wherein the flavor-producing article is a combustion-type flavor inhaler or a non-combustion-heating-type flavor inhaler.

40 ADVANTAGEOUS EFFECTS OF INVENTION

[0011] The present invention can provide an inexpensive method for manufacturing a tobacco-component-concentrated liquid, the method making concentration possible while adequately retaining the flavor component, and a tobaccocomponent-concentrated liquid manufactured by the method, as well as a method for manufacturing a flavor-producing

45 article including the method for manufacturing a tobacco-component-concentrated liquid, and a flavor-producing article manufactured by the method.

BRIEF DESCRIPTION OF DRAWINGS

50 [0012]

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Fig. 1 is a cross-sectional view of an example of a freeze concentration apparatus that can be used in a progressive freeze concentration method according to the present embodiment.

Fig. 2 is a cross-sectional view of an example of a combustion-type flavor inhaler according to the present embodiment.

Fig. 3 is a schematic diagram of an example of a non-combustion-heating-type flavor inhalation system according to the present embodiment, illustrating (a) a state before a non-combustion-heating-type flavor inhaler is inserted into a heating device and (b) a state in which the non-combustion-heating-type flavor inhaler is inserted into the heating device and is heated.

Fig. 4 is a chromatogram obtained by component analysis of tobacco-component-concentrated liquids according to Example 1 and Comparative Example 1.

Fig. 5 is a graph showing the total peak area in each RI range of the chromatogram of Fig. 4.

DESCRIPTION OF EMBODIMENTS

[Method for Manufacturing Tobacco-Component-Concentrated Liquid]

- 10 [0013] A method for manufacturing a tobacco-component-concentrated liquid according to the present embodiment includes the step of concentrating a liquid containing a tobacco component by a progressive freeze concentration method (hereinafter also referred to as a concentration process). The "freeze concentration method" is a method for cooling a solution to selectively solidify a solvent in the solution and physically separating and removing the solidified solvent to increase the concentration of the solution. The "freeze-drying method" includes sublimating and removing only a solvent
- ¹⁵ after the solution is entirely frozen and is different from the "freeze concentration method" in which only a solvent in the solution is selectively solidified and removed by cooling. In the method according to the present embodiment, a liquid containing a tobacco component is concentrated by a progressive freeze concentration method, which is one of the freeze concentration method. The "progressive freeze concentration method" is a method for forming a single large solidified solvent crystal by cooling to concentrate the solution.
- 20 [0014] In the method according to the present embodiment, the concentration operation performed at a low temperature can prevent or suppress the volatilization, dissipation, or change in quality of a flavor component contained in the liquid containing the tobacco component. Thus, the method can perform concentration while adequately retaining the flavor component. Concentration at a low temperature can also prevent or suppress contamination by microorganisms. Furthermore, selectively solidifying only the solvent and separating and removing the solidified solvent can sufficiently
- increase the concentration of the concentrated liquid. Furthermore, the latent heat required for a phase change is, for example, approximately one-seventh of an evaporative concentration method, and it is possible to save energy and reduce running costs. In the method according to the present embodiment, the liquid containing the tobacco component is concentrated by the freeze concentration method, particularly by the progressive freeze concentration method. This entails a lower cost than other freeze concentration methods, such as a suspension crystal method, and can also concentrate the liquid with high viscosity.
 - **[0015]** The method according to the present embodiment is not particularly limited as long as the method includes the concentration step, and preferably further includes the step of extracting a tobacco component in a tobacco raw material with a solvent to manufacture a liquid containing the tobacco component (hereinafter also referred to as a tobacco component extract manufacturing step) before the concentration step. The method preferably further includes the step
- ³⁵ of filtering the liquid containing the tobacco component to remove a solid (hereinafter also referred to as a filtration step) before the concentration step. When the method includes both the tobacco component extract manufacturing step and the filtration step, the filtration step can be performed after the tobacco component extract manufacturing step and before the concentration step. The method may further include another step other than these steps. Although each step is described below, the method according to the present embodiment is not limited to embodiments relating to these steps.
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(Tobacco Component Extract Manufacturing Step)

[0016] The method according to the present embodiment further includes the step of extracting a tobacco component in a tobacco raw material with a solvent to manufacture a liquid containing the tobacco component before the concentration step. Thus, the liquid containing the tobacco component is preferably a tobacco component extract obtained by extracting the tobacco component in the tobacco raw material with the solvent. This step allows the liquid containing the tobacco component extract) to be easily manufactured.

[0017] The tobacco raw material may include various types of tobacco. Examples thereof include flue-cured varieties, burley varieties, oriental varieties, native varieties, and other Nicotiana tabacum varieties and Nicotiana rustica varieties.

Although these varieties can be used alone, they can be used as a blend to generate a desired flavor. The details of the variety of tobacco are disclosed in "Tabako no jiten (Tobacco Dictionary), Tobacco Academic Studies Center, March 31, 2009".

[0018] The tobacco raw material preferably has a shape suitable for extraction of a tobacco component, for example, ground tobacco. When the tobacco raw material has a shape of ground tobacco, the tobacco raw material may have an average grain size of 10 μ m or less, for example.

[0019] Any solvent, such as water, ethanol, chloroform, or ethyl acetate, may be used for the extraction. These solvents may be used alone or in combination. Among these, the solvent is preferably water to easily control freeze concentration.[0020] The ratio of the mass of the tobacco raw material to the total mass of the solvent and the tobacco raw material

for the extraction preferably ranges from 5% to 20% by mass, more preferably 8% to 15% by mass, in terms of efficient extraction. The extraction temperature may be, for example, 20°C to 60°C, depending on the extracting solvent. The extraction time depends on the extracting solvent and the extraction temperature and may range from 1 to 3 hours, for example. The extraction can be performed, for example, by introducing the tobacco raw material and the extracting solvent into an extraction apparatus and stirring them.

- **[0021]** The "liquid containing a tobacco component" in the method according to the present embodiment is not limited to the tobacco component extract and may be, for example, a fraction obtained by distilling the tobacco raw material, or a compressed liquid of the tobacco raw material.
- 10 (Filtration Step)

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[0022] The method according to the present embodiment preferably further includes the step of filtering the liquid containing the tobacco component to remove a solid before the concentration step. The filtration step can remove a solid contained in the liquid containing the tobacco component, such as a protein or suspended matter. This can prevent

- or suppress the incorporation of the flavor component into the solidified solvent in the cooling step in the subsequent concentration step and consequently further increase the concentration of the flavor component in the concentrated liquid.
 [0023] The liquid containing the tobacco component can be filtered, for example, through a filter element, such as a filter cloth or a membrane filter. The filter element may have any mesh size, which may be, for example, 0.1 µm to 800 µm to mainly remove a fine powder of a tobacco raw material or another suspended matter. The filter element may have
- ²⁰ a mesh size in the range of 2 nm to 100 nm to mainly remove a protein. Two or more filter elements with different mesh sizes may be used for filtration.

(Concentration Step)

- ²⁵ **[0024]** The method according to the present embodiment includes the step of concentrating a liquid containing a tobacco component by a progressive freeze concentration method. A typical freeze concentration method may be a suspension crystal method. The suspension crystal method is a method for forming a large number of fine solidified solvent crystals by cooling to concentrate the solution. However, this method is only applied to large-scale continuous production due to high apparatus costs and requires a long residence time. In the method according to the present
- ³⁰ embodiment, therefore, the liquid containing the tobacco component is concentrated by the progressive freeze concentration method, which is a freeze concentration method. The progressive freeze concentration method is a method for forming a single large solidified solvent crystal by cooling to concentrate the solution. An apparatus to be used in this method is simple and versatile and can reduce the cost. Although the viscosity of a solution to be concentrated in the suspension crystal method is approximately 200 cP or less, the progressive freeze concentration method can perform sufficient concentration even at a viscosity of 200 cP or more.
- [0025] Fig. 1 illustrates an example of a freeze concentration apparatus that can be used for the progressive freeze concentration method in the present embodiment. A freeze concentration apparatus 1 illustrated in Fig. 1 has an impeller blade 4, and a refrigerant 2 is circulated around the outer circumference. A liquid 3 containing a tobacco component is introduced into the freeze concentration apparatus 1, and the refrigerant 2 can be set to a predetermined temperature
- 40 while stirring the liquid 3 containing the tobacco component with the impeller blade 4 to cool the liquid 3 to a predetermined temperature. With the passage of a predetermined time from the start of cooling, a layer of a solidified solvent 5 is formed on the inner surface of the freeze concentration apparatus 1, and the liquid 3 containing the tobacco component can be concentrated. In the freeze concentration apparatus 1, the liquid 3 containing the tobacco component can be cooled with stirring to reduce the inflow of a flavor component into the solidified solvent 5. Freeze concentration can be performed
- ⁴⁵ in a short time by controlling the temperature of the refrigerant 2 and controlling the temperature of the liquid 3 containing the tobacco component to a predetermined temperature. A freeze concentration apparatus that can be used in the present embodiment is not limited to the batch-type freeze concentration apparatus 1 illustrated in Fig. 1 and may be a flow-type freeze concentration apparatus, for example.
- [0026] The set temperature of the refrigerant 2 depends on the type of solvent contained in the liquid 3 containing the tobacco component. For example, when the solvent is water, the set temperature preferably ranges from -15°C to 0°C, more preferably -10.0°C to -3.0°C. At such a set temperature, the temperature of the liquid 3 containing the tobacco component ranges from -3.0°C to 0°C. When the temperature is within the above range, the inflow of a flavor component into the solidified solvent 5 (ice) can be further reduced, and the concentration operation can be performed in a short time. [0027] The stirring speed of the impeller blade 4 preferably ranges from 70 to 250 rpm, more preferably 100 to 180
- ⁵⁵ rpm. When the stirring speed is within such a range, the inflow of a flavor component into the solidified solvent 5 can be further reduced.

[0028] When freeze concentration is performed with a batch-type freeze concentration apparatus as illustrated in Fig. 1, the freeze concentration operation may be performed multiple times. For example, the liquid 3 containing the tobacco

component is introduced into the freeze concentration apparatus 1 illustrated in Fig. 1 and is cooled. After a predetermined time, the liquid 3 containing the tobacco component thus concentrated is taken out, and the solidified solvent 5 formed on the inner surface of the freeze concentration apparatus 1 is collected. The concentrated liquid 3 containing the tobacco component is again introduced into the freeze concentration apparatus 1 and is cooled to perform the concentration

- ⁵ operation again. The operation may be performed two or more times or three or more times. The operation can be performed multiple times to sufficiently increase the concentration ratio ((the mass of the liquid containing the tobacco component after concentration/the mass of the liquid containing the tobacco component before concentration) * 100)). The concentration ratio in the concentration step can be appropriately determined for a desired purpose and may range from, for example, 5% to 20%, preferably 7% to 15%.
- 10 [0029] In particular, the method according to the present embodiment can sufficiently retain the flavor component in the tobacco-component-concentrated liquid after the concentration step is performed. More specifically, when the tobacco-component-concentrated liquid manufactured by the method according to the present embodiment is subjected to headspace analysis, in a chromatogram obtained by gas chromatography-mass spectrometry (GC/MS), the ratio of the total area of peaks of a compound group with an RI of 2000 or less to the total area of all peaks (the ratio of a
- ¹⁵ compound group having 20 or less carbon atoms) is preferably 70% or more, more preferably 80% or more, still more preferably 90% or more, particularly preferably 95% or more. The upper limit of the range of the ratio may be, but is not limited to, 99.9% or less, for example. In the chromatogram, the ratio of the total area of peaks of a compound group with an RI in the range of 1000 to 2000 (the ratio of a compound group having 10 to 20 carbon atoms) is preferably 55% or more, more preferably 65% or more, still more preferably 70% or more. The upper limit of the range of the ratio may be.
- ²⁰ be, but is not limited to, 90% or less, for example. In the chromatogram, the ratio of the total area of peaks of a compound group with an RI of 1000 or less (the ratio of a compound group having 10 or less carbon atoms) is preferably 20% or more, more preferably 25% or more. The upper limit of the range of the ratio may be, but is not limited to, 50% or less, for example. The analytical conditions for the gas chromatography-mass spectrometry (GC/MS) are described below.
- 25 <Analytical Conditions>

Sampling rate: 2

[0030]

	-	Gas chromatography-mass spectrometry (GC/MS)
30		Apparatus: 7890B/5977B GC/MSD manufactured by Agilent Technologies
	-	GC conditions
		Column: HP-5MS UI (manufactured by Agilent Technologies)
		0.25 mm in inside diameter x 30 m in length, 0.25 μm in film thickness
35		Injection volume: 1 μl
		Injection mode: split (10:1)
		Inlet temperature: 270°C
		Septum purge flow rate: 5 ml/min
		Carrier gas: helium (He)
40		Column flow rate: 1 ml/min (constant flow rate mode)
		Oven temperature: 40°C (3 minutes) - 4°C/min - 280°C (20 minutes)
		Transfer line temperature: 280°C
	-	MS conditions
45		
		Solvent latency: 4 minutes
		Ionization method: electron impact ionization method (EI method), 70 eV
		Ion source temperature: 230°C
		Quadrupole temperature: 150°C
50		Measurement mode: scanning
		MS scan range: m/z 26 to 450
		Threshold: 50

⁵⁵ **[0031]** The peaks of the compound group with an RI of 2000 or less in the chromatogram are mainly assigned to a flavor component, and the ratio in the above range indicates that the tobacco-component-concentrated liquid contains a sufficient amount of flavor component. The RI represents a retention index and is more specifically calculated by a method described later.

[Tobacco-Component-Concentrated Liquid]

[0032] The tobacco-component-concentrated liquid according to the present embodiment is manufactured by the method for manufacturing a tobacco-component-concentrated liquid according to the present embodiment. The tobaccocomponent-concentrated liquid manufactured by the method according to the present embodiment can have a high flavor component content and a low solvent concentration (a high concentration ratio).

[0033] For example, when the tobacco raw material is flue-cured tobacco, with respect to the flavor component content, as described above, in a chromatogram obtained by the analysis method described above, the ratio of the total area of peaks of a compound group with an RI of 1000 or less to the total area of all peaks (the ratio of the total area of peaks

- 10 of a compound group having 10 or less carbon atoms) is preferably 20% or more. In such a case, the tobacco-componentconcentrated liquid contains a large amount of compound, such as 6-methyl-5-hepten-2-one. Furthermore, the ratio of the total area of peaks of a compound group with an RI of 2000 or less (the ratio of the total area of peaks of a compound group having 20 or less carbon atoms) is preferably 70% or more. A high content of a compound group with an RI of 2000 or less (a compound group having a carbon number of 20 or less) results in an appropriate amount and balance 15 of the flavor component.
 - [0034] With respect to the solvent concentration, for example, when the solvent is water, the water content of the tobacco-component-concentrated liquid preferably ranges from 20% to 90% by mass, more preferably 40% to 80% by mass. The water content is a value measured by Karl Fischer titration.
- 20 [Method for Manufacturing Flavor-Producing Article]

[0035] A method for manufacturing a flavor-producing article according to the present embodiment includes the steps of manufacturing a tobacco-component-concentrated liquid by the method according to the present embodiment (hereinafter referred to as a tobacco-component-concentrated liquid manufacturing step), applying the tobacco-component-

- 25 concentrated liquid to a substrate and drying the tobacco-component-concentrated liquid to manufacture a tobaccocomponent-containing substrate (hereinafter referred to as a tobacco-component-containing substrate manufacturing step), and manufacturing a flavor-producing article including the tobacco-component-containing substrate (hereinafter referred to as a flavor-producing article manufacturing step). In the method according to the present embodiment, the tobacco-component-concentrated liquid is manufactured by the method according to the present embodiment and is
- 30 used to manufacture the flavor-producing article. Thus, the flavor-producing article containing a predetermined amount of flavor component can be produced at low cost. The method according to the present embodiment is not particularly limited as long as the method includes the tobacco-component-concentrated liquid manufacturing step, the tobaccocomponent-containing substrate manufacturing step, and the flavor-producing article manufacturing step, and may further include another step other than these steps.
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(Tobacco-Component-Containing Substrate Manufacturing Step)

[0036] The method according to the present embodiment includes the step of applying the tobacco-component-concentrated liquid manufactured by the method according to the present embodiment to a substrate and drying the tobacco-40 component-concentrated liquid to manufacture a tobacco-component-containing substrate. The substrate may be, but is not limited to, a residue after extracting a tobacco component in a tobacco raw material with a solvent in the tobacco component extract manufacturing step, pulp, or the like. The tobacco-component-concentrated liquid can be applied to the substrate by coating, for example. The drying conditions after the tobacco-component-concentrated liquid is applied

- to the substrate are not particularly limited. For example, for hot-air drying, drying can be performed at 35°C to 100°C 45 until the final water content reaches a desired value. Furthermore, far-infrared radiation, middle-infrared radiation, or near-infrared radiation can be used to remove water. Infrared radiation drying can cause less vaporization and alteration due to heat than hot-air drying and can more successfully retain a tobacco component than hot-air drying. Furthermore, infrared radiation drying can advantageously selectively vaporize a component.
- [0037] When the flavor-producing article is a non-combustion-heating-type flavor inhaler, an aerosol-source material 50 that generates aerosol smoke upon heating may be further added to the substrate. The aerosol-source material may be of any type and can be an extract from various natural products and/or a constituent thereof selected according to the use. Specific examples of the aerosol-source material include, but are not limited to, polyhydric alcohols, such as glycerin, propylene glycol, sorbitol, xylitol, and erythritol, triacetin, 1,3-butanediol, and mixtures thereof. In addition to the tobacco-component-concentrated liquid and the aerosol-source material, a flavoring agent or the like may be further
- 55 added to the substrate.

(Flavor-Producing Article Manufacturing Step)

[0038] The method according to the present embodiment includes the step of manufacturing a flavor-producing article including the tobacco-component-containing substrate manufactured by the method. The flavor-producing article may be, for example, a combustion-type flavor inhaler, a non-combustion-heating-type flavor inhaler, or the like, as described later. The tobacco-component-containing substrate can be included, for example, in a tobacco-containing segment of the inhaler. The flavor-producing article can be manufactured by a known method.

[Flavor-Producing Article]

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[0039] The flavor-producing article according to the present embodiment is manufactured by the method for manufacturing a flavor-producing article according to the present embodiment. The flavor-producing article manufactured by the method for manufacturing a flavor-producing article according to the present embodiment can contain a desired amount of flavor component and is inexpensive. The flavor-producing article is a combustion-type flavor inhaler or a

15 non-combustion-heating-type flavor inhaler, for example.

(Combustion-Type Flavor Inhaler)

- [0040] Fig. 2 illustrates an example of a combustion-type flavor inhaler according to the present embodiment. As 20 illustrated in Fig. 2, a combustion-type flavor inhaler 6 includes a tobacco-containing segment 7 and a filter segment 8 adjacent to the tobacco-containing segment 7. The tobacco-containing segment 7 includes a tobacco filler 9 containing a tobacco-component-containing substrate to which the tobacco-component-concentrated liquid according to the present embodiment has been applied, and a wrapping paper 10 around the tobacco filler 9. The filter segment 8 may be any segment that functions as a general filter, and may be, for example, a tow made of synthetic fiber (also simply referred
- 25 to as a "tow") or a material, such as paper, processed into a cylindrical shape. The tobacco-containing segment 7 and the filter segment 8 are connected by a tipping paper member 11 wrapped on the tobacco-containing segment 7 and the filter segment 8. The tipping paper member 11 may have a vent hole in part of its outer circumference. The number of vent holes may be one or more, for example, 10 to 40. For a plurality of vent holes, the vent holes may be aligned in a circular shape on the outer circumference of the tipping paper member 11, for example. The plurality of vent holes
- 30 may be arranged at almost regular intervals. A vent hole allows air to be drawn through the vent hole into the filter segment 8 during inhalation. A product with a desired tar value can be designed by diluting the mainstream smoke with the outside air from a vent hole.

[0041] The user can enjoy the flavor of the tobacco by igniting the tip of the tobacco-containing segment 7 and putting a mouthpiece end of the filter segment 8 in his or her mouth for inhalation. The number of the filter segments 8 is not limited to one. For example, a plurality of filter segments with different functions may be connected.

(Non-Combustion-Heating-Type Flavor Inhaler)

[0042] The non-combustion-heating-type flavor inhaler according to the present embodiment may include, for example, 40 a tobacco-containing segment, a tubular cooling segment with a hole on the periphery, a center hole segment, and a filter segment. The non-combustion-heating-type flavor inhaler according to the present embodiment may have another segment, in addition to the tobacco-containing segment, the cooling segment, the center hole segment, and the filter segment.

- [0043] The non-combustion-heating-type flavor inhaler according to the present embodiment may have any axial 45 length and preferably has an axial length of 40 mm or more and 90 mm or less, more preferably 50 mm or more and 75 mm or less, still more preferably 50 mm or more and 60 mm or less. The non-combustion-heating-type flavor inhaler preferably has a circumferential length of 16 mm or more and 25 mm or less, more preferably 20 mm or more and 24 mm or less, still more preferably 21 mm or more and 23 mm or less. For example, the tobacco-containing segment has a length of 20 mm, the cooling segment has a length of 20 mm, the center hole segment has a length of 8 mm, and the
- 50 filter segment has a length of 7 mm. The length of the filter segment can be selected in the range of 4 mm or more and 10 mm or less. The airflow resistance of the filter segment is selected in the range of 15 mmH₂O/seg or more and 60 mmH₂O/seg or less per segment. The length of each segment can be appropriately changed according to the manufacturability, guality requirements, and the like. Only the filter segment on the downstream side of the cooling segment without the center hole segment can also function as a non-combustion-heating-type flavor inhaler.
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<Tobacco-Containing Segment>

[0044] The tobacco-containing segment may include a tobacco filler containing a tobacco-component-containing sub-

strate to which the tobacco-component-concentrated liquid according to the present embodiment has been applied, and a wrapping paper around the tobacco filler. The wrapping paper (hereinafter also referred to as a wrapper) may be filled with the tobacco filler by any method, for example, by wrapping the tobacco filler with the wrapper or by filling a tubular wrapper with the tobacco filler. When the shape of the tobacco-component-containing substrate has a longitudinal

⁵ direction, as in a rectangular shape, the tobacco-component-containing substrate may be filled such that the longitudinal direction is an unspecified direction in the wrapper or such that the longitudinal direction is an axial direction of the tobacco-containing segment or a direction perpendicular to the axial direction. Heating the tobacco-containing segment vaporizes a tobacco component (a flavor component), an aerosol-source material, and water contained in the tobacco filler, which are transferred to a mouthpiece segment by inhalation.

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<Cooling Segment>

[0045] The cooling segment may be composed of a tubular member. The tubular member may be, for example, a paper tube formed by processing thick paper into a cylindrical shape.

¹⁵ **[0046]** The cooling segment may have a total surface area of 300 mm²/mm or more and 1000 mm²/mm or less. This surface area is the surface area per length (mm) in the cooling segment airflow direction. The cooling segment preferably has a total surface area of 400 mm²/mm or more, more preferably 450 mm²/mm or more, and 600 mm²/mm or less, more preferably 550 mm²/mm or less.

[0047] It is desirable that the cooling segment have an internal structure with a large total surface area. Thus, in a preferred embodiment, the cooling segment may be formed of a sheet of a thin material that is wrinkled to form a channel and then pleated, gathered, and folded. More folds or pleats within a given volume of the element increase the total surface area of the cooling segment.

[0048] In some embodiments, the thickness of a constituent material of the cooling segment may be 5 μ m or more and 500 μ m or less, for example, 10 μ m or more and 250 μ m or less.

- **[0049]** The aerosol cooling element may be formed from a material with a specific surface area of 10 mm²/mg or more and 100 mm²/mg or less. In one embodiment, the specific surface area of a constituent material may be approximately 35 mm²/mg. The specific surface area can be determined in consideration of a material with a known width and thickness. For example, the material may be poly(lactic acid) with an average thickness of 50 μ m and a variation of ±2 μ m. When the material also has a known width of, for example, 200 mm or more and 250 mm or less, the specific surface area
- ³⁰ and density can be calculated.

[0050] The tubular member and a mouthpiece lining paper described later have a hole penetrating them. The hole allows the outside air to be introduced into the cooling segment during inhalation. This brings a vaporized aerosol component generated by heating the tobacco-containing segment into contact with the outside air, lowers the temperature of the vaporized aerosol component, liquefies the vaporized aerosol component, and forms an aerosol. The hole may

³⁵ have any diameter (full length), for example, of 0.5 mm or more and 1.5 mm or less. The number of holes may be, but is not limited to, one or two or more. For example, a plurality of holes may be provided on the periphery of the cooling segment.

[0051] The amount of outside air introduced through a hole is preferably 85% by volume or less, more preferably 80% by volume or less, of the volume of the whole gas inhaled by the user. When the amount of outside air is 85% by volume

40 or less, it is possible to sufficiently reduce the decrease in flavor due to dilution with the outside air. This is also referred to as a ventilation ratio. The lower limit of the ventilation ratio is preferably 55% by volume or more, more preferably 60% by volume or more, in terms of cooling performance.

[0052] The cooling segment preferably has less resistance to air passing through the tobacco-containing segment. The cooling segment preferably does not substantially affect the inhalation resistance of the non-combustion-heating-

- 45 type flavor inhaler. The resistance to draw (RTD) is the pressure required to push air through the total length of an object in a test at a flow rate of 17.5 ml/s at 22°C and 101 kPa (760 torr). RTD is typically expressed in mmH₂O and is determined in accordance with ISO 6565:2011. Thus, the pressure drop from the upstream end of the cooling segment to the downstream end of the cooling segment is preferably small. To achieve this, preferably, the longitudinal porosity is more than 50%, and the airflow path through the cooling segment is relatively unconstrained. The longitudinal porosity of the
- cooling segment may be defined by the ratio of the cross-sectional area of the material forming the cooling segment to the internal cross-sectional area of the cooling segment.
 [0053] In some embodiments, generated aerosol may be cooled by 10°C or more when inhaled by the user through the cooling segment. The temperature may be decreased by 15°C or more in another embodiment and by 20°C or more in still another embodiment.
- ⁵⁵ **[0054]** The cooling segment may be composed of a sheet material selected from the group including metal foils, polymer sheets, and substantially nonporous paper or thick paper. In one embodiment, the cooling segment may contain a sheet material selected from the group consisting of polyethylene, polypropylene, poly(vinyl chloride), poly(ethylene terephthalate), poly(lactic acid), cellulose acetate, and aluminum foil. A constituent material of the cooling segment may

be made from a biodegradable material, for example, nonporous paper, or a biodegradable polymer, such as poly(lactic acid), or a starch copolymer.

[0055] The airflow through the cooling segment preferably does not substantially deflect between adjacent segments. In other words, the airflow through the cooling segment preferably flows along the segment in the longitudinal direction

- without substantial radial deflection. In some embodiments, the cooling segment is formed from a low-porosity or substantially nonporous material, except for longitudinally extending channels. A material used to define or form a longitudinally extending channel, for example, a wrinkled or gathered sheet, has low porosity or is substantially nonporous.
 [0056] As described above, the cooling segment may include a sheet of an appropriate constituent material that is wrinkled, pleated, gathered, or folded. A cross-sectional profile of such an element may have randomly oriented channels.
- The cooling segment may be formed by another means. For example, the cooling segment may be formed from a bundle of longitudinally extending tubes. The cooling segment may be formed by extrusion, forming, lamination, injection, or shredding of an appropriate material.

[0057] The cooling segment may be formed, for example, by wrapping a pleated, gathered, or folded sheet material with a wrapping paper. In some embodiments, the cooling segment may include a sheet of a wrinkled material gathered into a rod shape and joined together by a wrapper, for example, a wrapping paper of filter paper.

[0058] The cooling segment may be formed in a rod shape with an axial length of, for example, 7 mm or more and 28 mm or less. For example, the cooling segment may have an axial length of 18 mm.

[0059] In some embodiments, the cooling segment is substantially circular in its axial cross-section and may have a diameter of 5 mm or more and 10 mm or less. For example, the cooling segment may have a diameter of approximately 7 mm.

<Center Hole Segment>

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[0060] The center hole segment is composed of a fill layer with one or more hollow portions and an inner plug wrapper (inner wrapping paper) covering the fill layer. For example, the center hole segment is composed of a second fill layer with a hollow portion and a second inner plug wrapper covering the second fill layer. The center hole segment functions to increase the strength of a mouthpiece segment. The second fill layer may be, for example, a rod with an inside diameter of φ1.0 mm or more and φ5.0 mm or less in which cellulose acetate fibers are densely packed and a plasticizer containing triacetin is added in an amount of 6% by mass or more and 20% by mass or less of the mass of cellulose acetate and

- ³⁰ is hardened. Due to the fibers with a high packing density in the second fill layer, air or aerosol flows only through the hollow portion during inhalation and rarely flows through the second fill layer. The second fill layer inside the center hole segment is a fiber fill layer, and the feeling of touch from the outside during use is less likely to cause discomfort to the user. The center hole segment may have no second inner plug wrapper and may maintain its shape by thermoforming.
- 35 <Filter Segment>

[0061] The filter segment may have any structure and may be composed of one or more fill layers. The outer side of the fill layer(s) may be wrapped with one or more wrapping papers. The airflow resistance per segment of the filter segment can be appropriately changed depending on the amount, material, and the like of filler in the filter segment.

- For example, when the filler is cellulose acetate fibers, increasing the amount of cellulose acetate fibers in the filter segment can increase the airflow resistance. When the filler is cellulose acetate fibers, the packing density of the cellulose acetate fibers may range from 0.13 to 0.18 g/cm³. The airflow resistance is a value measured with an airflow resistance measuring instrument (trade name: SODIMAX, manufactured by SODIM).
- **[0062]** The filter segment may have any circumferential length, which preferably ranges from 16 to 25 mm, more preferably 20 to 24 mm, still more preferably 21 to 23 mm. The axial length of the filter segment can be selected from 4 to 10 mm and is selected to have an airflow resistance in the range of 15 to 60 mmH₂O/seg. The filter segment preferably has an axial length in the range of 5 to 9 mm, more preferably 6 to 8 mm. The filter segment may have any cross-sectional shape, for example, a circular shape, an elliptical shape, a polygonal shape, or the like. A breakable capsule containing a flavoring agent, flavoring agent beads, or a flavoring agent may be added directly to the filter segment.
- ⁵⁰ **[0063]** The center hole segment and the filter segment can be connected with an outer plug wrapper (outer wrapping paper). The outer plug wrapper may be, for example, cylindrical paper. The tobacco-containing segment, the cooling segment, and the connected center hole segment and filter segment can be connected with a mouthpiece lining paper. These connections can be made, for example, by applying the inner surface of the mouthpiece lining paper with a glue, such as a vinyl acetate glue, putting the three segments on the paper, and wrapping the paper. These segments may
- ⁵⁵ be connected multiple times with a plurality of lining papers.

(Non-Combustion-Heating-Type Flavor Inhalation System)

[0064] A non-combustion-heating-type flavor inhalation system according to the present embodiment can include the non-combustion-heating-type flavor inhaler according to the present embodiment and a heating device for heating the tobacco-containing segment of the non-combustion-heating-type flavor inhaler. The non-combustion-heating-type flavor inhalation system according to the present embodiment may have another constituent, in addition to the non-combustion-heating-type flavor inhaler according to the present embodiment and the heating device.

[0065] Fig. 3 illustrates an example of the non-combustion-heating-type flavor inhalation system according to the present embodiment. The non-combustion-heating-type flavor inhalation system illustrated in Fig. 3 includes a noncombustion-heating-type flavor inhaler 12 according to the present embodiment and a heating device 13 for heating a tobacco-containing segment of the non-combustion-heating-type flavor inhaler 12 from the outside.

- **[0066]** Fig. 3(a) illustrates a state before the non-combustion-heating-type flavor inhaler 12 is inserted into the heating device 13, and Fig. 3(b) illustrates a state in which the non-combustion-heating-type flavor inhaler 12 is inserted into the heating device 13 and is heated. The heating device 13 illustrated in Fig. 3 includes a body 14, a heater 15, a metal
- ¹⁵ tube 16, a battery unit 17, and a control unit 18. The body 14 has a tubular recess 19. The heater 15 and the metal tube 16 are arranged on the inner side surface of the recess 19 at a position corresponding to the tobacco-containing segment of the non-combustion-heating-type flavor inhaler 12 inserted into the recess 19. The heater 15 may be an electrical resistance heater and is heated by an electric power supplied from the battery unit 17 according to an instruction from the control unit 18 for temperature control. Heat generated by the heater 15 is transferred to the tobacco-containing
- 20 segment of the non-combustion-heating-type flavor inhaler 12 through the metal tube 16 with high thermal conductivity. [0067] Although there is a space between the outer circumference of the non-combustion-heating-type flavor inhaler 12 and the inner circumference of the metal tube 16 in schematically illustrated Fig. 3(b), it is actually desirable that for efficient heat transfer there be no space between the outer circumference of the non-combustion-heating-type flavor inhaler 12 and the inner circumference of the metal tube 16. The heating device 13 heats the tobacco-containing segment
- of the non-combustion-heating-type flavor inhaler 12 from the outside but may heat it from the inside. [0068] The heating temperature by the heating device is preferably, but is not limited to, 400°C or less, more preferably 150°C or more and 400°C or less, still more preferably 200°C or more and 350°C or less. The heating temperature refers to the temperature of the heater of the heating device.
- 30 EXAMPLES

[0069] Although the present embodiments are described in more detail in the following examples, the present embodiments are not limited to these examples. Component analysis of a tobacco-component-concentrated liquid was performed by the following method.

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[Component Analysis of Tobacco-Component-Concentrated Liquid]

[0070] Tobacco-component-concentrated liquids manufactured by the examples and comparative examples were subjected to headspace analysis. First, 5 mL of a tobacco-component-concentrated liquid was heated at 60°C for 1 hour to adsorb a volatile component on an adsorbent. The adsorbent was Monotrap RCC 18 (trade name) manufactured by GL Sciences Inc. Extraction was performed by adding 300 μL of a mixed solvent of hexane: acetone = 1:1 (volume ratio) to the adsorbent. The resulting extract was analyzed by gas chromatography-mass spectrometry (GC/MS). 7890B manufactured by Agilent was used as a gas chromatograph, 5977B MSD (trade name) was used as a detector, and HP-5 ms UI (30m x 250 μm x 0.25 μm) was used as a column. Helium gas was used as a carrier gas for the gas

⁴⁵ chromatograph at a flow rate of 1.0 mL/min. The temperature was increased from 40°C to 280°C at a rate of 4°C/min and was held at 280°C for 20 minutes.

[0071] The retention index (RI) of a chromatogram obtained by the analysis was calculated by the following method. The retention index (RI) was calculated by a linear method using a n-alkane mixture ranging from n-hexane (C6, RI: 600) to n-pentatricontane (C35, RI: 3500). The n-alkane mixture used to calculate the retention index (RI) is not limited

50 to this. RI: 0 to 1000 corresponds to a compound group having 10 or less carbon atoms, RI: 1000 to 1500 corresponds to a compound group having 10 to 15 carbon atoms, RI: 1500 to 2000 corresponds to a compound group having 15 to 20 carbon atoms, and RI: 2000 to 2500 corresponds to a compound group having 20 to 25 carbon atoms.

[Example 1]

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(Tobacco Component Extract Manufacturing Step)

[0072] Tobacco leaves (flue-cured varieties) and water were mixed at a ratio of tobacco leaves: water = 1:10 (mass

ratio) and were stirred at 50°C for 2 hours to prepare a tobacco component extract.

(Filtration Step)

5 [0073] The tobacco component extract was filtered through a filter cloth (a nylon gyoza squeezing bag, mesh size: 0.45 mm) to remove fine particles in the tobacco component extract.

(Concentration Step)

- 10 [0074] The tobacco component extract after the filtration step was concentrated by a progressive freeze concentration method using a stirring type freeze concentration apparatus (trade name: PFC-M10, manufactured by Meiwa Co., Ltd.). More specifically, 10 kg of the tobacco component extract was put into a 12-L vessel and was concentrated to 7.145 kg at a brine temperature of -17°C with stirring at 120 rpm to prepare a first concentrated liquid (concentration ratio: 71.45%). At the same time, 2.855 kg of first ice was collected as an ice portion. Subsequently, 7.145 kg of the first concentrated
- 15 liquid was put into an 8-L vessel and was concentrated to 3.765 kg at a brine temperature of -17°C with stirring at 120 rpm to prepare a second concentrated liquid (concentration ratio: 37.65%). At the same time, 3.38 kg of second ice was collected as an ice portion. Furthermore, 3.765 kg of the second concentrated liquid was put into a 6-L vessel and was concentrated to 1.605 kg at a brine temperature of -17°C with stirring at 150 rpm to prepare a third concentrated liquid (tobacco-component-concentrated liquid) (concentration ratio: 16.05%). At the same time, 2.16 kg of third ice was
- 20 collected as an ice portion.

[0075] The tobacco-component-concentrated liquid was subjected to component analysis by the method described above. Fig. 4 is a chromatogram obtained by the component analysis. Fig. 5 shows the total peak area in each RI range in the chromatogram. Table 1 shows the ratio of the total peak area in each RI range.

25 [Comparative Example 1]

(Tobacco Component Extract Manufacturing Step, Filtration Step)

[0076] A tobacco component extract was prepared in the same manner as in Example 1 and was subjected to the 30 filtration step.

(Concentration Step)

[0077] The tobacco component extract after the filtration step was concentrated by an evaporative concentration 35 method. More specifically, 200 g of the tobacco component extract was concentrated to 32 g by heating at 40°C under a reduced pressure of 40 mmHg using a rotary evaporator (manufactured by Nihon BUCHI K.K.). The evaporation temperature was 34°C. Thus, a tobacco-component-concentrated liquid (concentration ratio: 16%) was prepared. [0078] The tobacco-component-concentrated liquid was subjected to component analysis by the method described above. Fig. 4 is a chromatogram obtained by the component analysis. Fig. 5 shows the total peak area in each RI range

40 in the chromatogram. Table 1 shows the ratio of the total peak area in each RI range.

45		Total peak area (-)		Total peak area ratio (%)		Peak area ratio (Example	
	RI	Example 1	Comparative example 1	Example 1	Comparative example 1	1/Comparative example 1)	
	0-1000	952563	821217	25.1	43.8	1.16	
	1000-1500	2285191	757135	60.1	40.4	3.02	
50	1500-2000	513192	249547	13.5	13.3	2.06	
	2000-2500	50474	44118	1.3	2.4	1.14	

Table 11

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[0079] As is clear from the chromatogram shown in Fig. 4, a comparison between Example 1 and Comparative Example 1 shows that the number of peaks of a component group with an RI of 2000 or less (a compound group having 20 or less carbon atoms) was larger and the intensity of each peak was also higher in Example 1 than in Comparative Example 1. [0080] Fig. 5 and Table 1 show that the amount of component particularly at an RI in the range of 1000 to 1500 in

Example 1 was approximately three times that in Comparative Example 1. The component group with an RI in this range may include phenolic compounds characterized by various flavors characteristic of tobacco. It was also found that the amount of component at an RI in the range of 1500 to 2000 in Example 1 was approximately twice that in Comparative Example 1. The component group with an RI in this range may include carotenoid decomposition products, such as megastigmatrienone, which is a flavor component of tobacco.

[0081] These results show that, while retaining a larger amount of flavor component in a liquid containing a tobacco component, such as in a tobacco component extract, concentration by the freeze concentration method can more sufficiently concentrate the liquid than by known evaporative concentration methods.

¹⁰ [Example 2, Comparative Example 2]

[0082] 50 g of shredded tobacco base sheets were scented by spraying with 5 g of the tobacco-component-concentrated liquid prepared in each of Example 1 and Comparative Example 1. The scented shredded sheets were dried in the open air at 35°C for 1 hour to prepare scented shredded sheets. A non-combustion-heating-type flavor inhaler containing the

- ¹⁵ scented shredded sheets in a tobacco-containing segment was prepared. The tobacco-containing segment of the inhaler was heated from the outside and was inhaled to evaluate the flavor. The non-combustion-heating-type flavor inhaler (Example 2) containing the scented shredded sheets of Example 1 more satisfactorily had the intrinsic flavor of tobacco than the non-combustion-heating-type flavor inhaler (Comparative Example 2) containing the scented shredded sheets of Comparative Example 1.
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REFERENCE SIGNS LIST

[0083]

- ²⁵ 1 freeze concentration apparatus
 - 2 refrigerant
 - 3 liquid containing a tobacco component
 - 4 impeller blade
 - 5 solidified solvent
 - 6 combustion-type flavor inhaler
 - 7 tobacco-containing segment
 - 8 filter segment
 - 9 tobacco filler
 - 10 wrapping paper
- 35 11 tipping paper member
 - 12 non-combustion-heating-type flavor inhaler
 - 13 heating device
 - 14 body
 - 15 heater
 - 16 metal tube
 - 17 battery unit
 - 18 control unit
 - 19 recess

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Claims

- 1. A method for manufacturing a tobacco-component-concentrated liquid, comprising the step of concentrating a liquid containing a tobacco component by a progressive freeze concentration method.
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- 2. The method according to claim 1, further comprising the step of extracting the tobacco component in a tobacco raw material with a solvent to manufacture the liquid containing the tobacco component before the step of concentrating the liquid containing the tobacco component by the progressive freeze concentration method.
- 55 **3.** The method according to claim 2, wherein the solvent contains water
 - 4. The method according to any one of claims 1 to 3, further comprising the step of filtering the liquid containing the tobacco component to remove a solid before the step of concentrating the liquid containing the tobacco component

by the progressive freeze concentration method.

- 5. The method according to any one of Claims 1 to 4, wherein a total area of peaks of a compound group with an RI of 2000 or less is 70% or more of a total area of all peaks in a chromatogram obtained by gas chromatographymass spectrometry (GC/MS) when the tobacco-component-concentrated liquid is subjected to headspace analysis.
- 6. A tobacco-component-concentrated liquid manufactured by the method according to any one of claims 1 to 5.
- 7. A method for manufacturing a flavor-producing article, comprising the steps of:
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manufacturing a tobacco-component-concentrated liquid by the method according to any one of claims 1 to 5; applying the tobacco-component-concentrated liquid to a substrate and drying the tobacco-component-concentrated liquid to manufacture a tobacco-component-containing substrate; and manufacturing a flavor-producing article including the tobacco-component-containing substrate.

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- 8. A flavor-producing article manufactured by the method according to claim 7.
- **9.** The flavor-producing article according to claim 8, wherein the flavor-producing article is a combustion-type flavor inhaler or a non-combustion-heating-type flavor inhaler.

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Fig. 1

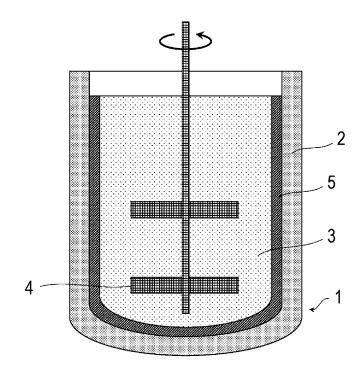


Fig. 2

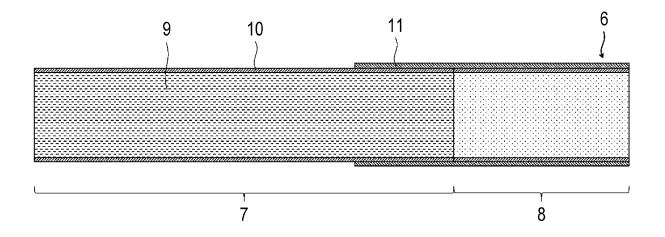
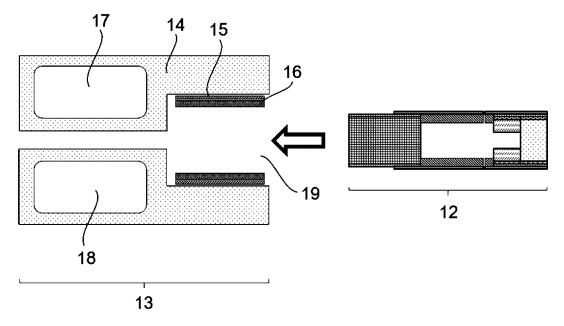
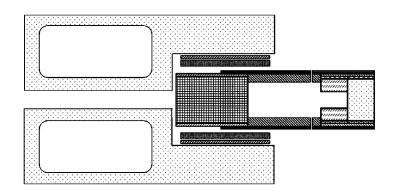


Fig. 3

(a)



(b)



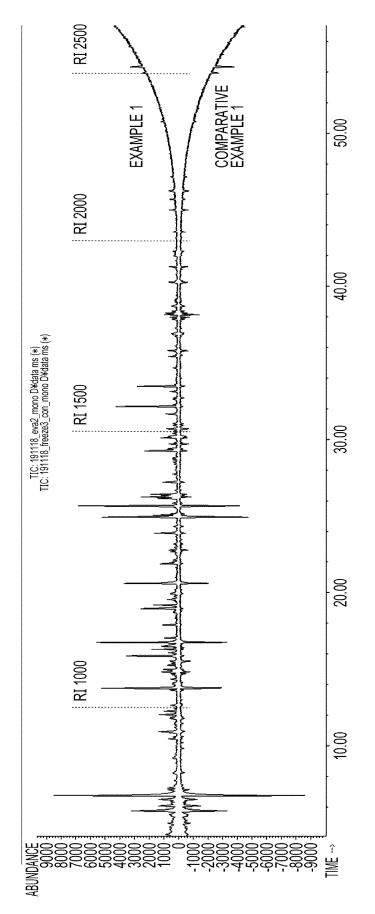
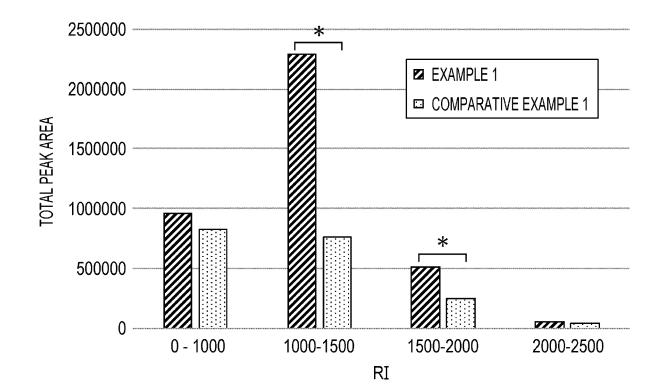


FIG. 4

Fig. 5



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