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(71) Applicant: **Japan Tobacco Inc.**
Tokyo 105-6927 (JP)

(72) Inventors:

- **CHIDA, Masahiro**
Tokyo 130-8603 (JP)
- **MIZUTANI, Masashi**
Tokyo 130-8603 (JP)
- **MATSUMOTO, Yuichi**
Tokyo 130-8603 (JP)
- **TSURUOKA, Naoya**
Tokyo 130-8603 (JP)

(74) Representative: **Hoffmann Eitle**

Patent- und Rechtsanwälte PartmbB
Arabellastraße 30
81925 München (DE)

(54) **TOBACCO EXTRACT CONTAINING TOBACCO TERPENES, AND METHOD FOR MANUFACTURING SAID TOBACCO EXTRACT**

(57) Provided is a method for producing a tobacco extract containing a tobacco terpene, the method including:

a process 1 that includes preparing a raw material derived from tobacco;

a process 2 that includes subjecting the raw material to solid-liquid extraction with an aprotic medium-polar solvent containing a hetero element;

a process 3 that includes recovering an organic phase from the process;

a process 4 that includes adding a protic polar solvent or an aprotic medium-polar solvent to an extract obtained by removing the solvent from the organic phase, to precipitate or disperse a solid content; and

a process 5 that includes removing the solid.

EP 4 245 155 A1

Description

TECHNICAL FIELD

5 **[0001]** The present invention relates to a tobacco extract containing tobacco terpenes and a method for producing the same, and more particularly to a tobacco extract containing sesquiterpene or diterpene and a method for producing the same.

BACKGROUND ART

10 **[0002]** The aroma components of tobacco leaves are aromas mainly composed of decomposition products derived from pigments generated at the time of maturation or in the drying process, that is, carotenoid decomposition products such as ionone and megastigmatrienone, and leaf surface resin components such as sesquiterpene and diterpene. Most of these aroma components are hydrophobic. In order to utilize these aroma components, various extraction methods have been studied. For example, many examples using liquefied carbon dioxide gas or supercritical carbon dioxide gas have been studied by taking advantage of the characteristics of a target component (PTLs 1 and 2). On the other hand, raw materials expanded with liquefied carbon dioxide gas have been studied as one of the raw materials for cigarettes, and the process is very similar to the extraction process using carbon dioxide gas described above. Therefore, a method has been studied in which a part of the expansion process is applied in order to utilize the aroma components (PTLs 3 and 4). For example, PTL 3 discloses an apparatus including: an extraction container that brings carbon dioxide in a supercritical state into contact with a tobacco raw material to dissolve a tobacco component in the carbon dioxide; a separation container that is connected to the extraction container and separates and recovers a fat-soluble ingredient of the tobacco component from the tobacco component dissolved in the carbon dioxide; a circulation path that circulates the carbon dioxide in a supercritical state between the extraction container and an absorption container that stores pure water while purifying the carbon dioxide with a purification layer made of activated carbon, and absorbs a water-soluble portion of the tobacco component in the pure water; and a recovery container that recovers pure water having absorbed the water-soluble portion of the tobacco component from the absorption container as absorption water. The fat-soluble ingredient of the tobacco component and the absorption water obtained from the apparatus are used for producing flavor. In addition, PTL 4 discloses a method in which a tobacco component contained in vaporized carbon dioxide that is exhausted by heating after impregnating a tobacco raw material with liquefied carbon dioxide is heated from 150°C to 400°C to obtain heated aroma. This method is very unique in that reductone is obtained. However, it is suggested that even unnecessary components derived from heating, as shown in PTL 5 and the like, may be generated.

CITATION LIST

PATENT LITERATURE

[0003]

40 PTL 1: International Publication No. WO 2007/029264
 PTL 2: GB Patent No. 2173985
 PTL 3: International Publication No. WO 2007/119790
 PTL 4: International Publication No. WO 2016/051334A1
 PTL 5: Japanese Patent Application Publication No. 2016-526921

SUMMARY OF INVENTION

TECHNICAL PROBLEM

50 **[0004]** In the conventional method, it has not been easy to efficiently obtain a flavor component from a raw material derived from tobacco by a simple method. Therefore, an object of the present invention is to provide a method for efficiently producing a component useful as a flavoring agent from a raw material derived from tobacco by a simple method.

SOLUTION TO PROBLEM

55 **[0005]** The inventors have found that the above problem can be solved by using, as a raw material, a waste including tobacco stem or a solid extract discharged simultaneously with separation of carbon dioxide gas in the expansion treatment of tobacco. That is, the above problem is solved by the following present invention.

Embodiment 1

[0006] A method for producing a tobacco extract containing a tobacco terpene, the method including:

- 5 a process 1 that includes preparing a raw material derived from tobacco;
a process 2 that includes subjecting the raw material to solid-liquid extraction with an aprotic medium-polar solvent containing a hetero element;
a process 3 that includes recovering an organic phase from the process;
10 a process 4 that includes adding a protic polar solvent or an aprotic medium-polar solvent to an extract obtained by removing the solvent from the organic phase, to precipitate or disperse a solid content; and
a process 5 that includes removing the solid.

Embodiment 2

- 15 **[0007]** The production method according to embodiment 1, in which a boiling point of the aprotic medium-polar solvent used in the process 2 is 80°C or lower.

Embodiment 3

- 20 **[0008]** The production method according to embodiments 1 and 2, in which the aprotic medium-polar solvent is selected from the group consisting of ethyl acetate, butyl butyrate, ethyl butyrate, dichloromethane, chloroform, and a combination thereof.

Embodiment 4

- 25 **[0009]** The production method according to any one of embodiments 1 to 3, in which the aprotic medium-polar solvent is ethyl acetate.

Embodiment 5

- 30 **[0010]** The production method according to any one of embodiments 1 to 4, in which the process 2 further includes subjecting an organic phase obtained by solid-liquid extraction to extraction with water or an acid aqueous solution, and acid used for the acid aqueous solution is an inorganic acid or an organic acid.

35 Embodiment 6

[0011] The method for producing a tobacco extract according to any one of embodiments 1 to 5, in which the acid is selected from the group consisting of sulfuric acid, citric acid, oxalic acid, and a combination thereof.

40 Embodiment 7

[0012] The production method according to any one of embodiments 1 to 6, in which the process 2 further includes subjecting an organic phase obtained by solid-liquid extraction to extraction with water or an acid aqueous solution, and a pH of the acid aqueous solution is 4 or less.

45 Embodiment 8

[0013] The production method according to any one of embodiments 1 to 7, in which the protic polar solvent used in the process 4 is ethanol.

50 Embodiment 9

[0014] The production method according to any one of embodiments 1 to 7, in which the aprotic medium-polar solvent used in the process 4 is benzyl alcohol.

55 Embodiment 10

[0015] The production method according to any one of embodiments 1 to 9, in which the process 4 includes adding

the solvent to the extract to form a solution once, and then precipitating a solid content.

Embodiment 11

- 5 **[0016]** The production method according to any one of embodiments 1 to 9, in which the process 4 includes adding the solvent to the extract, to obtain a dispersion in which a solid content is dispersed.

Embodiment 12

- 10 **[0017]** The production method according to embodiment 11, further including promoting precipitation of the solid content.

Embodiment 13

- 15 **[0018]** The production method according to any one of embodiments 1 to 12, in which a concentration of the extract in the solution or the dispersion prepared in the process 4 is 5 to 20 wt%.

Embodiment 14

- 20 **[0019]** A tobacco extract obtained by the method according to embodiments 1 to 13.

Embodiment 15

- 25 **[0020]** A tobacco flavoring agent containing the tobacco extract according to embodiment 14.

Embodiment 16

- 30 **[0021]** A tobacco material containing the tobacco flavoring agent according to embodiment 15.

Embodiment 17

[0022] The tobacco material according to embodiment 16, in which the tobacco material is a tobacco sheet or a shredded tobacco.

35 Embodiment 18

[0023] A tobacco rod part containing the tobacco material according to embodiment 16 or 17.

Embodiment 19

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[0024] A tobacco flavor inhalation article including the tobacco rod part according to embodiment 18.

Embodiment 20

- 45 **[0025]** A non-combustion heating type tobacco flavor inhalation article or a non-combustion non-heating type tobacco flavor inhalation article including the tobacco rod part according to embodiment 18.

Embodiment 21

- 50 **[0026]** A smokeless tobacco containing the tobacco extract according to embodiment 14.

BRIEF DESCRIPTION OF DRAWINGS

[0027]

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FIG. 1 is a GC/MS total ion chromatogram of a hexane solution obtained in Experimental Example 1. In FIGs. 1 to 6, the vertical lines indicate the retention time at which RI is 1,600 to 2,500, and the arrow indicates the peak of CBT (cembratrienediol).

FIG. 2 is a GC/MS total ion chromatogram of a chloroform solution obtained in Experimental Example 1.

FIG. 3 is a GC/MS total ion chromatogram of an ethyl acetate solution obtained in Experimental Example 1.

FIG. 4 is a GC/MS total ion chromatogram of an acetone solution obtained in Comparative Example 1.

FIG. 5 is a GC/MS total ion chromatogram of a methanol solution obtained in Comparative Example 1.

FIG. 6 is GC/MS total ion chromatograms of an alcohol preparation (upper stage) obtained in Example 4 and an alcohol preparation (lower stage) from shreds before expansion, obtained in the same manner as in Example 4.

FIG. 7 is a GC/MS total ion chromatogram of an alcohol preparation obtained in Example 5. The vertical lines indicate the retention times at which RI is 800, 1,600, 2,500, and 3,500.

FIG. 8 is a GC/MS total ion chromatogram of a benzyl alcohol preparation obtained in Example 10. The vertical lines indicate the retention times at which RI is 800, 1,600, 2,500, and 3,500.

FIG. 9 is a view illustrating one embodiment of a non-combustion heating type tobacco flavor inhalation article.

FIG. 10 is a view illustrating one embodiment of a non-combustion heating type tobacco flavor inhalation system.

FIG. 11 is a view illustrating one embodiment of a non-combustion non-heating type tobacco flavor inhalation article.

FIG. 12 is a view illustrating one embodiment of a tobacco capsule.

FIG. 13 is a view illustrating one embodiment of a power supply unit.

FIG. 14 is a cross-sectional view of one embodiment of a cartridge.

FIG. 15 is a view illustrating an internal structure of one embodiment of the cartridge.

FIG. 16 is a view illustrating an outline of an expansion treatment process.

FIG. 17 is a GC/MS total ion chromatogram of a preparation (flue-cured variety raw material) obtained in Example 12.

FIG. 18 is a GC/MS total ion chromatogram of a preparation (Burley variety raw material) obtained in Example 12.

DESCRIPTION OF EMBODIMENTS

[0028] Hereinafter, the present invention will be described in detail. In the present invention, the expression "X to Y" includes the end values X and Y.

1. Production method

[0029] The production method of the present invention includes the following processes:

Process 1: preparing a raw material derived from tobacco;

Process 2: subjecting the raw material to solid-liquid extraction with an aprotic solvent;

Process 3: recovering an organic phase from the process;

Process 4: adding a protic polar solvent or an aprotic medium-polar solvent to an extract obtained by removing the solvent from the organic phase, to precipitate or disperse a solid content; and

Process 5: removing the solid content.

(1) Process 1

[0030] In this process, a raw material derived from tobacco is prepared. The raw material derived from tobacco is a raw material derived from plants of the genus *Tobacco*. Examples thereof include tobacco raw materials such as tobacco leaves, aged tobacco leaves, shredded tobacco, or tobacco powder, and processed products or wastes obtained by subjecting the tobacco raw materials to processing. The tobacco leaves are a generic term for harvested tobacco leaves that have not undergone aging. One embodiment of aging includes curing. The shredded tobacco is obtained by shredding aged tobacco leaves or the like into a predetermined size. The tobacco powder is obtained by pulverizing tobacco leaves or the like.

[0031] In the present invention, it is preferable to prepare a discharged solid obtained in the expansion treatment process of the tobacco raw material. The expansion treatment refers to a treatment of impregnating a tobacco raw material with a liquid and rapidly vaporizing the liquid to increase the bulk of the tobacco material (see FIG. 16). In the present invention, a discharged solid discharged by a known expansion treatment can be used. As described above, the tobacco raw material is a tobacco leaf, a shredded tobacco, or a tobacco powder. In the present invention, it is preferable to use, as the discharged solid, solid fat-soluble ingredients obtained when an expansion treatment is performed with supercritical carbon dioxide and carbon dioxide is separated from supercritical carbon dioxide containing tobacco components, as described in PTL 3. This is because the discharged solid obtained from the expansion treatment using supercritical carbon dioxide contains a large amount of tobacco terpenes. The tobacco terpenes are preferably sesquiterpene or diterpene (hereinafter, also simply referred to as "terpenes"). In addition, since the expansion treatment process includes a process of threshing and shredding tobacco leaves as a raw material at the initial stage, tobacco fine powder and stems as a small amount of waste shreds are mixed into the tobacco raw material. When the raw

material is subjected to an expansion treatment, after the expansion, light expanded shreds, heavy stems, and non-expanded shreds are discharged together with carbon dioxide gas, and become discharged solids. The discharged solids are the above-described solid fat-soluble ingredients, or solids of expanded shreds, stems, and non-expanded shreds, or mixtures thereof accordingly. Hereinafter, an embodiment in which the discharged solid is used as a raw material derived from tobacco will be described as an example.

(2) Process 2

[0032] In this process, the discharged solid is subjected to solid-liquid extraction with an aprotic solvent. The aprotic solvent is a solvent that is hardly soluble in water and can form an organic phase separated from an aqueous phase because it does not have a proton to be dissociated. Examples of the aprotic solvent include esters such as ethyl acetate, butyl butyrate, and ethyl butyrate; halogenated hydrocarbons such as dichloromethane and chloroform; ketones such as acetone; nitriles such as acetonitrile; and hydrocarbons such as hexane. Among them, an aprotic medium-polar solvent containing a hetero element, such as an ester or a halogenated hydrocarbon is preferable from the viewpoint of efficiently extracting target terpenes. In addition, from the viewpoint of easy removal in a subsequent process, solvents having a boiling point of 80°C or lower are preferable. Therefore, as the solvent, ethyl acetate, butyl butyrate, ethyl butyrate, dichloromethane, and chloroform are preferable, ethyl acetate, butyl butyrate, and ethyl butyrate are more preferable, and ethyl acetate is still more preferable. In this process, the target terpenes are transferred to the aprotic solvent (organic phase).

[0033] The aprotic solvent can be classified into low-polar solvents and medium-polar solvents depending on their polarity. In one embodiment, based on the octanol/water partition coefficient (Kow), the aprotic medium-polar solvent is defined as a solvent exhibiting a positive value of log Kow of 2 or less, and the aprotic low-polar solvent is defined as a solvent exhibiting a value of log Kow of greater than 2 and 4 or less. In the present invention, the aprotic medium-polar solvent is preferable. Kow is defined as the ratio of the concentration of the target compound (solvent) dissolved in the octanol phase to the concentration of the target compound dissolved in the aqueous phase in a two-phase system of octanol and water. Kow is measured at room temperature.

$$Kow = \text{concentration of octanol phase} / \text{concentration of aqueous phase}$$

[0034] In one embodiment, the solvent used in the present invention is classified as exemplified below.

[Table 1]

Category	Compound	log Kow	Polarity
Protic	Propylene glycol	-1.4	High polarity
	Methanol	-0.77	
	Ethanol	-0.24	
	Acetone	-0.24	
Aprotic	Ethyl acetate	0.73	Medium polarity
	Diethyl ether	0.89	
	Benzyl alcohol	1.1	
	Cumyl alcohol*	1.95	
	Chloroform	1.97	
	Benzene	2.13	Nonpolarity
	Hexane	3.9	
Source: www.chem.ucta.edu/~bacher/Generat/30BL/tips/sotvent.html			
*Source: MSDS			

[0035] In this process, the discharged solid may be subjected to extraction with an aprotic solvent and water or an acid aqueous solution. This is because nicotine in the discharged solid can be transferred to the aqueous phase by water. Since nicotine in the discharged solid can be transferred, in the form of a salt, to the aqueous phase by acid, the water preferably contains an acid, that is, the water is preferably an acid aqueous solution. As the acid, an inorganic acid or an organic acid can be used, but sulfuric acid, citric acid, or oxalic acid is preferable from the viewpoint of stability of the nicotine salt and the like. The pH of the acid aqueous solution is preferably 4 or less, and more preferably 3 or

less. When the pH exceeds 4, nicotine may not be sufficiently extracted. The lower limit of the pH is not limited, but is preferably 2 or more. The temperature at which this process is carried out is not limited, but is preferably 10 to 35°C, and more preferably 20 to 30°C. In this process, it is preferable that the discharged solid be subjected to solid-liquid extraction with an aprotic solvent, and then the organic phase be subjected to liquid-liquid extraction with the water or acid aqueous solution. In the solid-liquid extraction, an insoluble solid content may be removed by filtration or the like.

(3) Process 3

[0036] In this process, the organic phase obtained in the process 2 is recovered. The organic phase is the organic phase obtained by the solid-liquid extraction or the organic phase obtained by the liquid-liquid extraction. The recovery method is not limited, and can be performed using, for example, a separatory funnel. If necessary, the aqueous phase may be washed with an aprotic solvent, and the washed solvent may be added to the organic phase. In this way, a solution of a tobacco extract containing terpenes can be obtained.

(4) Process 4

[0037] In this process, first, the aprotic solvent is removed from the organic phase to obtain the extract. The method for removing the solvent is not limited, and for example, an evaporator can be used. A protic polar solvent or an aprotic medium-polar solvent is added to the extract to precipitate or disperse a solid content. The protic solvent is an organic solvent having a dissociable proton, and examples thereof include aliphatic alcohols such as methanol, ethanol, and propanol; glycols such as propylene glycol; and ketones such as acetone. Examples of the aprotic medium-polar solvent used in this process include aromatic alcohols such as benzyl alcohol, phenylethyl alcohol, and cumyl alcohol; esters such as ethyl acetate; ethers such as diethyl ether; and chlorinated hydrocarbons such as chloroform. In one embodiment, the protic polar solvent is defined as a solvent exhibiting a negative value of log Kow. The aprotic medium-polar solvent used in the process 4 may be the same as, but preferably different from, the aprotic solvent used in the process 2. The aprotic medium-polar solvent used in the process 4 is preferably an aromatic alcohol.

[0038] The protic polar solvent (preferably, aliphatic alcohol) phase obtained in this process contains target terpenes. Since these terpenes have a retention index (hereinafter, also simply referred to as "RI") of 1,600 to 2,500 in gas chromatography, the protic polar solvent phase contains components having an RI of 1,600 to 2,500. The protic polar solvent phase preferably does not contain components having an RI of more than 2,500 and 3,500 or less. This is because the components having an RI of more than 2,500 and 3,500 or less are mainly higher hydrocarbons, and produce an undesirable smoking flavor when heated to 160 to 250°C. Therefore, the components contained in the protic polar solvent phase are suitable for a tobacco flavor inhalation article heated to 160 to 250°C, and more suitable for a tobacco flavor inhalation article heated to 160°C or higher and lower than 220°C. RI is an index obtained by standardizing the retention time of gas chromatography using a standard substance. In the present invention, RI refers to an index obtained by a linear method using the retention time of n-paraffin as a scale.

[0039] The aprotic medium-polar solvent (preferably, aromatic alcohol) phase obtained in this process contains target terpenes. Since the terpenes have an RI of 1,600 to 2,500, the aprotic medium-polar solvent phase contains components having an RI of 1,600 to 2,500. Further, the aprotic medium-polar solvent phase preferably contains components having an RI of more than 2,500 and 3,500 or less, that is, contains components having an RI of 1,600 to 3,500. The components having an RI of more than 2,500 and 3,500 or less are nonpolar, dissolve aroma components better, and have a function as a retention agent. Therefore, when the components are heated to 220 to 280°C, the flavor components are volatilized to give soft and smooth smoking flavor. Accordingly, the tobacco extract obtained using the solvent is suitable for a tobacco flavor inhalation article heated to 220 to 280°C, and more suitable for a tobacco flavor inhalation article heated to higher than 250°C and 280°C or lower.

[0040] The protic polar solvent is preferably ethanol, and the aprotic medium-polar solvent is benzyl alcohol from the viewpoint of imparting excellent smoking flavor, safety, and the like.

[0041] In this process, the solid content is precipitated or dispersed by adding the solvent. This process specifically includes an embodiment in which the solvent is added to the extract to form a solution once, and then a solid content is precipitated, and an embodiment in which the solvent is added to the extract to quickly obtain a dispersion in which a solid content is dispersed. In the latter embodiment, it is preferable to further promote the precipitation of the solid content. The precipitation method or the method for promoting precipitation is not limited, and examples thereof include allowing the system to stand and centrifuging. At this time, the temperature of the system is preferably -10 to 10°C. Setting such a temperature prevents the target terpenes from deteriorating. The amount of the solvent is not limited, but is such an amount that the concentration of the extract in the liquid is preferably 5 to 20 wt% and more preferably 8 to 15 wt%, from the viewpoint of efficiently obtaining the precipitate.

(5) Process 5

[0042] In this process, the solid is removed. The removal method is not limited and can be carried out by filtration or decantation.

2. Tobacco extract

[0043] The tobacco extract obtained by the production method (hereinafter, also referred to as "tobacco extract of the present invention") contains the terpenes. Since the terpenes impart smoking flavor of tobacco, the tobacco extract of the present invention is useful as a tobacco flavoring agent. The tobacco flavoring agent is a tobacco extract in one embodiment, and contains a tobacco extract and other components in another embodiment. As described above, the tobacco extract obtained using the aprotic medium-polar solvent in the process 4 is suitable for a tobacco flavor inhalation article heated to 220 to 280°C, and the tobacco extract obtained using the protic polar solvent is suitable for a tobacco flavor inhalation article heated to 160 to 250°C. Further, the tobacco extract obtained using the acid aqueous solution in the process 2 does not contain alkaloids such as nicotine or the amount thereof is extremely small. The tobacco extract of the present invention can also be a tobacco flavoring agent (preparation) prepared by dissolving the tobacco extract in ethanol, benzyl alcohol or propylene glycol. In the preparation, the concentration of the tobacco extract of the present invention can be about 10 to 30 wt%. A tobacco flavor preparation is excellent in handleability because it can be sprayed onto an object or impregnated into an object. The amount of the preparation added is preferably 50 to 200 ppm by weight based on the weight of the tobacco material in the case of combustion tobacco, is preferably 0.2 to 0.75 wt% based on the weight of the tobacco material in the case of non-combustion type tobacco, and is preferably 50 to 200 ppm by weight based on the weight of the base liquid in the case of liquid-heated tobacco.

3. Tobacco material

[0044] A tobacco flavoring agent containing the tobacco extract of the present invention (hereinafter also referred to as "tobacco flavoring agent of the present invention") is useful as an additive to a tobacco material. Examples of the tobacco material include tobacco materials such as a tobacco sheet, a shredded tobacco, a cigarette paper, and a polysaccharide sheet. The tobacco material to which the tobacco flavoring agent of the present invention has been added is also referred to as "tobacco material of the present invention".

(1) Tobacco sheet

[0045] The tobacco sheet is a sheet obtained by forming a composition containing aged tobacco leaves and the like into a sheet shape. The aged tobacco leaves used for the tobacco sheet are not particularly limited, and examples thereof include laminas and stems separated from tobacco leaves by stemming. The aged tobacco leaves refer to tobacco leaves that have undergone processing such as curing and long-term storage in a warehouse or the like. In the present invention, the "sheet" refers to a material having a pair of substantially parallel main surfaces and side surfaces. The tobacco sheet can be formed by a known method such as a papermaking method, a casting method, or a rolling method. Details of various tobacco sheets formed by such methods are disclosed in "Encyclopedia of Tobacco, Tobacco Academic Studies Center, March, 31, 2009". The embodiment in which the tobacco flavoring agent of the present invention is added to the tobacco sheet is not limited.

[0046] For example, a solution of the tobacco flavoring agent of the present invention is prepared, and the solution may be sprayed onto or impregnated into a completed tobacco sheet. Alternatively, the tobacco flavoring agent of the present invention may be added to the tobacco sheet when the tobacco sheet is formed. For example, the papermaking method includes: separating aged tobacco leaves into a water extract and a residue through extraction of water-soluble components from the tobacco leaves; subjecting a mixture of the residue that has been defibrated and pulp to papermaking; and adding a concentrate of the water extract to a sheet obtained by papermaking. In this method, the tobacco flavoring agent of the present invention can be added to the water extract. The casting method includes: mixing water, pulp, a binder, a pulverized product of aged tobacco to prepare a mixture; and subjecting the mixture to casting. In this method, the tobacco flavoring agent of the present invention can be added to the mixture. The rolling method includes: mixing water, pulp, a binder, and a pulverized product of aged tobacco to prepare a mixture; supplying the mixture to a plurality of rolling rollers; and performing rolling. In this method, the tobacco flavoring agent of the present invention can be added to the mixture.

[0047] Further, as described in International Publication No. WO 2014/104078, a pulverized product of aged tobacco and a binder are mixed to prepare a mixture, the mixture is sandwiched between nonwoven fabrics, and the laminate is formed into a certain shape by heat welding, whereby a nonwoven fabric-shaped tobacco sheet can be obtained. In this method, the tobacco flavoring agent of the present invention can be added to the mixture.

[0048] The tobacco sheet may contain an aerosol-generating base material. The type of the aerosol-generating base material is not particularly limited, and various extracted substances from natural products or their constituent components can be selected depending on the application. Specific examples of the aerosol-generating base material may include polyhydric alcohols such as glycerin, propylene glycol, sorbitol, xylitol, and erythritol, triacetin, 1,3-butanediol, and mixtures thereof. The content of the aerosol-generating base material can be adjusted to various amounts depending on the form utilized in the tobacco product. For example, when the aerosol-generating base material is contained in the tobacco sheet, the content thereof is usually 5 wt% or more, preferably 10 wt% or more, and more preferably 15 wt% or more, and is usually 50 wt% or less, preferably 40 wt% or less, and more preferably 25 wt% or less, based on the total weight of the tobacco sheet, from the viewpoint of obtaining good flavor.

(2) Shredded tobacco

[0049] Examples of the shredded tobacco include those obtained by shredding aged tobacco leaves into a predetermined size, those obtained by shredding the tobacco sheet into a predetermined size, and those obtained by mixing these. The size of the shredded tobacco is not limited, and examples thereof include those having a width of 0.5 to 2.0 mm and a length of 3 to 10 mm. The shredded tobacco having such a size is preferable in an embodiment in which an object to be filled described later is filled with the shredded tobacco. In addition, examples of the shredded tobacco include strand type shreds prepared by shredding processed tobacco leaves so as to have a width of 0.5 to 2.0 mm and a length longer than that of the shredded tobacco described above, preferably about the same length as that of the cigarette paper. The tobacco flavoring agent of the present invention may be added to a shredded tobacco, or may be added to a raw material before shredding.

[0050] The shredded tobacco may contain the aerosol-generating base material. When the aerosol-generating base material is contained in the shredded tobacco, the content thereof is usually 5 wt% or more, preferably 10 wt% or more, and more preferably 15 wt% or more, and is usually 50 wt% or less, preferably 40 wt% or less, and more preferably 25 wt% or less, based on the weight of the shredded tobacco, from the viewpoint of generating a sufficient amount of aerosol and obtaining good flavor.

(3) Cigarette paper

[0051] A cigarette paper containing the tobacco flavoring agent can be prepared by, for example, spraying the tobacco flavoring agent of the present invention onto the cigarette paper, or impregnating the cigarette paper with the tobacco flavoring agent. Examples of the cigarette paper include paper containing pulp as a main component. Besides pulps made of wood pulp such as softwood pulp and hardwood pulp, the pulp may be a pulp obtained by mixing, with wood pulp, non-wood pulp generally used for a cigarette paper for tobacco articles, such as flax pulp, hemp pulp, sisal pulp, and esparto. These pulps may be used alone, or a plurality of types may be used in combination at any ratio. Further, the cigarette paper may be composed of one sheet, or may be composed of a plurality of sheets or more. The cigarette paper may be used in the form of a wrapped member in which a tobacco raw material such as shredded tobacco is wrapped with the cigarette paper. The cigarette paper can also be used as a material (for example, tipping paper) for wrapping the wrapped member together with other members such as a cooling member and a filter member. As the pulp, a chemical pulp obtained by a kraft cooking method, an acidic-neutral-alkali sulfite cooking method, a soda cooking method or the like, a ground pulp, a chemiground pulp, a thermomechanical pulp and the like can be used.

(4) Polysaccharide sheet

[0052] The polysaccharide sheet is a sheet containing a polysaccharide as a main component, and the polysaccharide sheet can contain the tobacco flavoring agent of the present invention. The flavor inhalation article using the polysaccharide sheet containing the tobacco flavoring agent of the present invention can release sufficient flavor. Examples of the polysaccharide include carrageenan, agar, gellan gum, tamarind gum, psyllium seed gum, konjac glucomannan, carrageenan, locust bean gum, guar gum, agar, xanthan gum, gellan gum, tamarind gum, tara gum, konjac glucomannan, starch, cassia gum, and psyllium seed gum.

[0053] The polysaccharide sheet containing the tobacco flavoring agent of the present invention can be used for a combustion type tobacco flavor inhalation article and a non-combustion type tobacco flavor inhalation article. In the former embodiment, for example, the polysaccharide sheet as disclosed in Japanese Patent No. 5481574 can be used. In this embodiment, the content of the tobacco flavoring agent of the present invention can be preferably 10 wt% or more, more preferably 18 wt% or more, still more preferably 60 wt% or more, and particularly preferably 70 wt% or more, based on the weight of the sheet. The polysaccharide sheet can be prepared by a method in which a polysaccharide and water are mixed and heated to prepare an aqueous solution of a polysaccharide, a flavor and an emulsifier are added to the aqueous solution, and the mixture is kneaded and emulsified. As the emulsifier, a known emulsifier can

be used.

[0054] In the latter embodiment, the polysaccharide sheet as described in PCT/JP 2019/20136 can be used. In this embodiment, agar is particularly preferably used as the polysaccharide. The content of the agar is preferably 10 to 50 wt%, more preferably 15 to 45 wt% based on the weight of the sheet. In addition, the content of the tobacco flavoring agent of the present invention in the polysaccharide sheet can be 35 to 80 wt% based on the weight of the sheet.

[0055] In this embodiment, it is preferable to use a saccharide compound selected from the group consisting of sugars and sugar alcohols. Examples of the "sugar" include glucose, sucrose, fructose, xylose, galactose, mannose, maltose, trehalose, lactose, and raffinose. Examples of the "sugar alcohol" include sorbitol which is an alcohol obtained by reducing the carbonyl group of a sugar to a hydroxyl group. The content of the compound is preferably 10 wt% or more, more preferably 10 to 500 wt%, still more preferably 10 to 300 wt%, and still more preferably 10 to 200 wt%, based on the weight of the agar. In this embodiment, an emulsifier is preferably used. As the emulsifier, a known emulsifier can be used, and the content thereof is preferably 0.5 to 10 wt% and more preferably 1.0 to 8.0 wt% based on the weight of the agar.

[0056] The polysaccharide sheet in this embodiment can be produced by kneading raw materials containing agar, a saccharide compound, a flavor, and an emulsifier in water to prepare a raw material slurry, extending the raw material slurry on a base material, and drying the slurry.

4. Tobacco flavor inhalation article

[0057] In the present invention, the "flavor inhalation article" refers to an article for a user to inhale flavor. Among flavor inhalation articles, those having tobacco or a component derived from the tobacco are referred to as "tobacco flavor inhalation article". The tobacco flavor inhalation article is roughly classified into "combustion type tobacco flavor inhalation articles" (also simply referred to as "smoking article") that generate flavor by combustion, and "non-combustion type tobacco flavor inhalation articles" that generate flavor without combustion. Further, the non-combustion type tobacco flavor inhalation article is roughly classified into "non-combustion heating type tobacco flavor inhalation articles" that generate flavor by heating and "non-combustion non-heating type tobacco flavor inhalation articles" that generate flavor without heating. The tobacco flavoring agent of the present invention is suitable for the non-combustion heating type tobacco flavor inhalation article or the non-combustion non-heating type tobacco flavor inhalation article. In addition, a combination of a device (a heating apparatus, an atomizing apparatus, or the like) for generating aerosol and the non-combustion heating type tobacco flavor inhalation article is also particularly referred to as a non-combustion heating type tobacco flavor inhalation system.

(1) Non-combustion heating type tobacco flavor inhalation article

[0058] FIG. 9 illustrates one embodiment of the non-combustion heating type tobacco flavor inhalation article. As illustrated in the drawing, a non-combustion heating type tobacco flavor inhalation article 20 includes a tobacco rod part 20A, a cylindrical cooling part 20B having perforations on the periphery thereof, and a filter part 20C. The non-combustion heating type tobacco flavor inhalation article 20 may have other members. The axial length of the non-combustion heating type tobacco flavor inhalation article 20 is not limited, but is preferably 40 to 90 mm, more preferably 50 to 75 mm, and still more preferably 50 to 60 mm or less. In addition, the circumferential length of the non-combustion heating type tobacco flavor inhalation article 20 is preferably 16 to 25 mm, more preferably 20 to 24 mm, and still more preferably 21 to 23 mm. For example, an embodiment can be exemplified in which the length of the tobacco rod part 20A is 20 mm, the length of the cooling part 20B is 20 mm, and the length of the filter part 20C is 7 mm. The length of each member can be appropriately changed according to production suitability, required quality, and the like. FIG. 9 illustrates an embodiment in which a first segment 25 is disposed, but only a second segment 26 may be disposed downstream of the cooling part 20B without disposing the first segment 25.

1) Tobacco rod part 20A

[0059] In the tobacco rod part 20A, as a tobacco filler 21, a shredded tobacco or a tobacco sheet containing the tobacco flavoring agent of the present invention can be used. A method for filling the tobacco filler 21 into a cigarette paper 22 is not particularly limited, but for example, the tobacco filler 21 may be wrapped with the cigarette paper 22, or the tobacco filler 21 may be filled into the cylindrical cigarette paper 22. When the shape of the tobacco has a longitudinal direction like a rectangular shape, the tobacco filler may be filled so that each longitudinal direction is an unspecified direction in the cigarette paper 22, or may be filled so as to be aligned in the axial direction of the tobacco rod part 20A or aligned in a direction orthogonal thereto. As the cigarette paper 22, a cigarette paper containing the tobacco flavoring agent of the present invention described above can also be used. When the tobacco rod part 20A is heated, the tobacco component, the aerosol-generating base material and water contained in the tobacco filler 21 are vaporized and provided

for inhalation.

2) Cooling part 20B

[0060] The cooling part 20B is preferably formed of a cylindrical member. The cylindrical member may be, for example, a paper tube 23 obtained by processing a cardboard into a cylindrical shape. The cooling part 20B may also be formed by a sheet of thin material that is wrinkled, then creased, gathered, or folded to form a channel. As such a material, for example, a sheet material selected from the group consisting of polyethylene, polypropylene, polyvinyl chloride, polyethylene terephthalate, polylactic acid, cellulose acetate, and aluminum foil can be used. The total surface area of the cooling part 20B is appropriately prepared in consideration of cooling efficiency, and can be set to, for example, 300 to 1,000 mm²/mm. The cooling part 20B is preferably provided with perforations 24. Due to the presence of the perforations 24, outside air is introduced into the cooling part 20B at the time of inhalation. As a result, the aerosol vaporization component generated by heating the tobacco rod part 21A comes into contact with the outside air, and the temperature thereof is lowered, so that the aerosol is liquefied and formed. The diameter (length across the perforation) of the perforation 24 is not particularly limited, but may be, for example, 0.5 to 1.5 mm. The number of perforations 24 is not particularly limited, and may be one or two or more. For example, a plurality of perforations 24 may be provided on the periphery of the cooling part 20B.

[0061] The cooling part 20B can have a rod shape having an axial length of, for example, 7 to 28 mm. The axial length of the cooling part 20B can be set to, for example, 18 mm. The cooling part 20B has a substantially circular axial cross-section, and can have a diameter of 5 to 10 mm. The diameter of the cooling part can be, for example, about 7 mm.

3) Filter part 20C

[0062] The configuration of the filter part 20C is not particularly limited, but may be composed of one or a plurality of filling layers. The outside of the filling layer may be wrapped with one or more sheets of cigarette paper. The air-flow resistance of the filter part 20C can be appropriately changed depending on the amount, material, and the like of the filler filled in the filter part 20C. For example, when the filler is cellulose acetate fibers, the air-flow resistance can be increased by increasing the amount of cellulose acetate fibers filled in the filter part 20C. When the filler is cellulose acetate fibers, the filling density of the cellulose acetate fibers can be 0.13 to 0.18 g/cm³. The air-flow resistance is a value measured by an air-flow resistance measuring device (trade name: SODIMAX, manufactured by SODIM).

[0063] The circumferential length of the filter part 20C is not particularly limited, but is preferably 16 to 25 mm, more preferably 20 to 24 mm, and still more preferably 21 to 23 mm. The axial length of the filter part 20C (horizontal direction in FIG. 9) can be selected to be 4 to 10 mm so that the air-flow resistance thereof is 15 to 60 mmH₂O/seg. The axial length of the filter part 20C is preferably 5 to 9 mm, and more preferably 6 to 8 mm. The shape of the cross section of the filter part 20C is not particularly limited, and can be, for example, a circle, an ellipse, a polygon, or the like. Further, a breakable capsule containing a flavor, flavor beads, and a flavor may be directly added to the filter part 20C.

[0064] The filter part 20C may include a center hole part as the first segment 25. The center hole part is composed of a first filling layer 25a having one or a plurality of hollow portions, and an inner plug wrapper (inner cigarette paper) 25b covering the filling layer. The center hole part has a function of increasing the strength of a mouthpiece part. The center hole part may not have the inner plug wrapper 25b, and the shape of the center hole part may be maintained by thermal molding. The filter part 20C may include the second segment 26. The second segment 26 is composed of a second filling layer 26a and an inner plug wrapper (inner cigarette paper) 26b covering the filling layer. The second filling layer 26a can be, for example, a rod having an inner diameter ϕ of 5.0 to ϕ 1.0 mm, made by filling the cellulose acetate fibers densely and then curing, wherein a plasticizer containing triacetin of 6 to 20 wt% (based on the weight of cellulose acetate) has been added to the cellulose acetate fibers. Since the second filling layer has a high filling density of fibers, air and aerosol flow only through the hollow portion at the time of inhalation, and hardly flow in the second filling layer. Since the second filling layer inside the center hole segment is a fiber filling layer, the touch feeling from the outside at the time of use hardly causes discomfort to the user.

[0065] The first filling layer 25b and the second filling layer 26a are connected by an outer plug wrapper (outer cigarette paper) 27. The outer plug wrapper 27 can be, for example, cylindrical paper. The tobacco rod part 20A, the cooling part 20B, and the first filling layer 25b and second filling layer 26a, which have been connected, are connected by a mouthpiece lining paper 28. The connection of these members can be made by, for example, applying a glue such as a vinyl acetate-based glue to the inner surface of the mouthpiece lining paper 28, and wrapping the mouthpiece lining paper 28 around the three members. These members may be connected by a plurality of times with a plurality of sheets of lining papers.

5) Non-combustion heating type tobacco flavor inhalation system

[0066] A combination of a non-combustion heating type tobacco flavor inhalation article and a heating device for

generating aerosol is particularly also referred to as a non-combustion heating type tobacco flavor inhalation system. An example of the system is illustrated in FIG. 10. In the drawing, the non-combustion heating type tobacco flavor inhalation system includes the non-combustion heating type tobacco flavor inhalation article 20 and a heating device 10 that heats the tobacco rod part 20A from the outside.

[0067] The heating device 10 includes a body 11, a heater 12, a metal tube 13, a battery unit 14, and a control unit 15. The body 11 has a cylindrical recess 16, and the heater 12 and the metal tube 13 are disposed at positions corresponding to the tobacco rod part 20A to be inserted into the recess. The heater 13 can be a heater by electric resistance, and power is supplied from the battery unit 14 according to an instruction from the control unit 15 that performs temperature control, and the heater 12 is heated. The heat generated from the heater 12 is transferred to the tobacco rod part 20A through the metal tube 13 having high thermal conductivity. In the drawing, an embodiment is illustrated in which the heating device 10 heats the tobacco rod part 20A from the outside, but the heating device may heat the tobacco rod part 20A from the inside. The heating temperature of the heating device 10 is not particularly limited, but is preferably 400°C or lower, more preferably 150 to 400°C, and still more preferably 200 to 350°C. The heating temperature indicates the temperature of the heater of the heating device 10. In particular, in the heating device 10, when the tobacco rod part contains the tobacco extract obtained using the protic polar solvent in the process 4, the tobacco rod part can be preferably heated to 160 to 250°C. In addition, in the heating device 10, when the tobacco rod part contains the tobacco extract obtained using the aprotic medium-polar solvent in the process 4, the tobacco rod part is preferably heated to 220 to 280°C.

(3) Non-combustion non-heating type tobacco flavor inhalation article

[0068] FIG. 11 illustrates one embodiment of the non-combustion non-heating type tobacco flavor inhalation article. A non-combustion non-heating type tobacco flavor inhalation article 30 includes a power supply unit 30D, a cartridge 30E, and a tobacco capsule 30F. The non-combustion non-heating type tobacco flavor inhalation article 30 has a shape extending from a non-inhalation end u (upstream) toward an inhalation end d (downstream). The cartridge 30E is detachable from the power supply unit 30D. The tobacco capsule 30F is detachable from the cartridge 30E.

1) Tobacco capsule

[0069] FIG. 12 illustrates an example of the tobacco capsule 30F. As shown in the drawing, the tobacco capsule 30F is a tobacco rod part, and has a flavor source 300 therein. The flavor source 300 contains the tobacco material of the present invention. The tobacco capsule 30F is connected to the cartridge 30E. Specifically, a part of the tobacco capsule 30F is accommodated in the cartridge 30E.

[0070] The tobacco capsule 30F includes a container 310 that accommodates the flavor source 300, a mesh body 320, a nonwoven fabric 330, and a cap 340. The aerosol atomized by an atomizer 220 to be described later is introduced into the container 310 through the mesh body 320 and comes into contact with the flavor source 300, whereby flavor is imparted to the aerosol. Thereafter, the aerosol is inhaled into the user through the nonwoven fabric 330. As described above, in the non-combustion non-heating type tobacco flavor inhalation article 30, flavor can be imparted to aerosol without heating the flavor source 300. In addition, substantially no aerosol is generated from the flavor source 300.

[0071] In the flow direction of aerosol, the length of the tobacco capsule 30F (container 310) is preferably 40 mm or less, and more preferably 25 mm or less. In the flow direction of aerosol, the length is preferably 1 mm or more, and more preferably 5 mm or more. In the direction orthogonal to the flow direction of aerosol, the maximum length of the container 310 of the tobacco capsule 30F (container 310) is preferably 20 mm or less, and more preferably 10 mm or less. In addition, in the direction orthogonal to the flow direction of aerosol, the maximum length of the tobacco capsule 30F (container 310) is preferably 1 mm or more, and more preferably 3 mm or more.

[0072] The flavor source 300 containing tobacco is composed of raw material pieces that impart flavor to aerosol. The lower limit of the size of the raw material piece is preferably 0.2 to 1.2 mm, and more preferably 0.2 to 0.7 mm. As the size of the raw material piece constituting the flavor source 300 decreases, the specific surface area increases, so that the smoking flavor components are easily released. As the raw material pieces constituting the flavor source 300, a shredded tobacco which is the tobacco material of the present invention, a formed body obtained by forming the tobacco raw material of the present invention into particles, or the like can be used. The flavor source 300 may contain a natural flavor of plants (for example, mint, herb, and the like) other than tobacco and menthol, a synthetic flavor, fruit juice, a taste material, a plant active ingredient, and the like. Examples of the taste material include materials exhibiting sweetness, sourness, saltiness, umami, bitterness, astringency taste, body taste, pungency, harsh taste, astringency, and the like. Examples of the material exhibiting sweetness include saccharides, sugar alcohols, and sweeteners. Examples of the saccharide include monosaccharides, disaccharides, oligosaccharides, and polysaccharides. Examples of the sweetener include natural sweeteners and synthetic sweeteners.

[0073] The raw material pieces are obtained, for example, by sieving in accordance with JIS Z 8801 using a stainless steel sieve in accordance with JIS Z 8815. For example, the raw material pieces are sieved over 20 minutes using a

stainless steel sieve having a mesh opening of 0.71 mm by a dry type mechanical shaking method, to obtain raw material pieces which have passed through the stainless steel sieve having a mesh opening of 0.71 mm. Subsequently, the raw material pieces are sieved over 20 minutes using a stainless steel sieve having a mesh opening of 0.212 mm by a dry type mechanical shaking method, to remove raw material pieces which have passed through the stainless steel sieve having a mesh opening of 0.212 mm. That is, the raw material pieces constituting the flavor source 300 are raw material pieces that have passed through a stainless steel sieve (mesh opening = 0.71 mm) defining the upper limit and have not passed through a stainless steel sieve (mesh opening = 0.212 mm) defining the lower limit. Therefore, the lower limit of the size of the raw material piece constituting the flavor source 300 is defined by the mesh opening of the stainless steel sieve that defines the lower limit. The upper limit of the size of the raw material piece constituting the flavor source 300 is defined by the mesh opening of the stainless steel sieve that defines the upper limit.

[0074] The filling amount of the flavor source 300 to be accommodated in the container 310 is preferably 300 mg or more, and more preferably 350 mg or more, from the viewpoint of increasing the volatilization amount of nicotine during smoking.

2) Power supply unit

[0075] An example of the power supply unit 30D is illustrated in FIG. 13. The power supply unit 30D includes a battery 110. The battery 110 may be a disposable battery or a rechargeable battery. The initial value of the output voltage of the battery 110 is preferably in a range of 1.2 V or more and 4.2 V or less. The battery capacity of the battery 110 is preferably in a range of 100 mAh or more and 1,000 mAh or less.

3) Cartridge

[0076] An example of the cartridge 30E is illustrated in FIGs. 14 and 15. FIG. 14 is a cross-sectional view of an example of the cartridge 30E, and FIG. 15 is a view illustrating an internal structure of the cartridge 30E. The cartridge 30E has a reservoir 210, an atomizer 220, a flow path forming body 230, an outer frame 240, and an end cap 250. The cartridge 30D has a first flow path 200X disposed downstream of the atomizer 220 as an aerosol flow path.

[0077] The reservoir 210 stores an aerosol source 200. The reservoir 210 is located around the flow path forming body 230 in a cross section orthogonal to the flow direction of aerosol (direction from the non-inhalation end to the inhalation end (upstream to downstream)). The reservoir 210 is located in a gap between the flow path forming body 230 and the outer frame 240. The reservoir 210 is made of, for example, a porous body such as a resin web or cotton. Further, the reservoir 210 may be composed of a tank that accommodates the liquid aerosol source 200. Examples of the aerosol source 200 include glycerin and propylene glycol.

[0078] The atomizer 220 atomizes the aerosol source 200 without combustion by the electric power supplied from the battery 110. The atomizer 220 is formed of heating wires (coils) wound at a predetermined pitch. The atomizer 220 is preferably formed of heating wires having a resistance value in a range of 1.0 to 3.0 Ω . The predetermined pitch is equal to or larger than a value at which the heating wires do not contact each other, and is preferably a smaller value. The predetermined pitch is preferably, for example, 0.40 mm or less. The predetermined pitch is preferably constant to stabilize the atomization of the aerosol source 200. The predetermined pitch is an interval between the centers of heating wires adjacent to each other.

[0079] The flow path forming body 230 has a cylindrical shape and forms the first flow path 200X extending along the flow direction of aerosol. The outer frame 240 has a cylindrical shape and accommodates the flow path forming body 230. The outer frame 240 extends downstream of the end cap 250 and accommodates a part of the tobacco capsule 30F. The end cap 250 is a cap that closes the gap between the flow path forming body 230 and the outer frame 240 from the downstream side. The end cap 250 suppresses a situation in which the aerosol source 200 stored in the reservoir 210 leaks to the tobacco capsule 30E side.

5. Smokeless tobacco

[0080] The smokeless tobacco is a product that contains a flavor source and allows the user to taste a flavor derived from the flavor source by directly taking the product in the nasal cavity or oral cavity. As the flavor source contained in the smokeless tobacco, the tobacco material of the present invention can be used. As the smokeless tobacco, snuff tobacco and chewing tobacco are known.

EXAMPLES

[Experimental Example 1] Selection of solvent for extracting active ingredient from solid

[0081] A discharged solid obtained from the expansion treatment process of a tobacco raw material using supercritical carbon dioxide was prepared. Specifically, the treatment as shown in FIG. 16 was performed to impregnate a tobacco raw material with carbon dioxide in a supercritical state, the tobacco raw material in a dry ice state was taken out, and then the tobacco raw material was air-dried at once to remove carbon dioxide. A discharged solid (Dust) resulting from solidification of gum components and shredded fine powder separated from high-temperature carbon dioxide gas (Tail gas) and carbon dioxide discharged at that time was obtained as the discharged solid.

[0082] About 5 g of the discharged solid was weighed in a 100 ml screw tube, and 50 ml of an organic solvent was added and mixed well, and then the mixture was allowed to stand at room temperature for a whole day and night. As the organic solvent, n-hexane, chloroform, and ethyl acetate were used. The mixed solution was filtered with filter paper, and a small amount of anhydrous sodium sulfate was added to the filtrate (extract) for dehydration. Then, the resultant was filtered with filter paper again to remove the organic solvent under reduced pressure. The same solvent as that used in the extraction was added to the obtained dried solid (yield: 3.4% of n-hexane, 7.8% of chloroform, 15.6% of ethyl acetate) to dissolve the dried solid, thus obtaining a solution having a concentration of the dried solid of 4 wt%. The solution was subjected to GC/MS analysis under the following conditions.

Gas chromatography with mass spectrometer (GC/MS)

Apparatus: 7890A/5975C GC/MSD manufactured by Agilent Technologies, Inc.

GC conditions

Column: HP-5MS (manufactured by Agilent Technologies, Inc.)

Inner diameter 0.25 mm × length 30 m, film thickness: 0.25 μm

Injection volume: 1 μl

Injection mode: split (10 : 1)

Inlet temperature: 270°C

Septum purge flow rate: 5 ml/min

Carrier gas: helium (He)

Column flow rate: 1 ml/min (constant flow mode)

Oven temperature: 40°C (3 min), temperature raised to 280°C at 4°C/min, 280°C (20 min)

Transfer line temperature: 280°C

MS conditions

Solvent waiting time: 4 min

Ionization method: electron impact ionization (EI method), 70 eV

Ion source temperature: 230°C

Quadrupole temperature: 150°C

Measurement mode: scan

MS scan range: m/z 26 to 450

Threshold: 50

Sampling rate: 2

[Comparative Example 1]

[0083] Extraction was performed in the same manner as in Experimental Example 1 except that acetone and methanol were used as solvents. The yield was 24.7% when acetone was used and 28.9% when methanol was used.

[0084] FIGs. 1 to 5 show GC/MS total ion chromatograms obtained by analysis of the n-hexane solution, the chloroform solution, the ethyl acetate solution, the acetone solution, and the methanol solution. The chromatograms in FIGs. 1 to 5 show that solvent extraction with n-hexane, chloroform, or ethyl acetate (FIGs. 1 to 3) results in a large peak area of the component group (sesquiterpene and diterpenes) having a retention index (RI) of 1,600 to 2,500. On the other hand, the chromatograms show that solvent extraction with acetone or methanol (FIGs. 4 and 5) results in a small peak area of the same component group. Among three types of solvents that allow efficient extraction of the desired same component group, ethyl acetate having the highest yield from the solid was found to be preferable as the extraction solvent. The chromatogram showed that extraction with chloroform resulted in possibility of remaining of trace amount of halogen in the extract.

[Experimental Example 2] Removal of nicotine

[0085] First, 150 g of the discharged solid was weighed, and put into a 2,500 ml sealed stainless steel container. Next, 1,500 ml of ethyl acetate (for high-performance liquid chromatography, FUJIFILM Wako Pure Chemical Corporation) was added to the discharged solid, and extraction was then performed for 3 hours in a warm bath at 40°C under hermetically stirring. After the extraction, the ethyl acetate solution and the extraction residue were separated with a stainless steel mesh having a mesh opening of 250 μm to obtain about 1,400 ml of an ethyl acetate solution. Then, 300 ml of the obtained ethyl acetate solution and 500 ml of each of diluted sulfuric acid waters adjusted to pH 5.0, pH 4.0, pH 3.0, and pH 2.0, respectively were placed in a 1,000 ml separatory funnel, and the mixture was subjected to liquid-liquid extraction to recover an organic phase. The solvent was removed from the organic phase under reduced pressure with a rotary evaporator. Ethyl acetate was added to the resulting concentrate so that the concentration of the dried solid was 4 wt%. The resulting solution was subjected to GC/MS analysis under the conditions shown in Experimental Example 1 to quantify alkaloids such as nicotine. The table below shows each peak area ratio with respect to all peaks represented on the total ion chromatogram. The peaks of respective alkaloids were separately confirmed with their characteristic single ion (for example, nicotine is m/z 84). The results showed that alkaloids mainly composed of nicotine were removed by extraction in an acidic region of pH of 4.0 or less.

[Table 2]

	GC/MS TIC area%			
alkaloid	pH 5.0	pH 4.0	pH 3.0	pH 2.0
nicotine	1.6	trace	n.d.	n.d.
nornicotine	0.1	n.d.	n.d.	n.d.
myosmine	0.3	n.d.	n.d.	n.d.
nicotyrine	0.1	n.d.	n.d.	n.d.
nicotine-N-oxide	n.d.	n.d.	n.d.	n.d.
anabasine	n.d.	n.d.	n.d.	n.d.
anatabine	n.d.	n.d.	n.d.	n.d.
cotinine	n.d.	n.d.	n.d.	n.d.

[Example 3] Removal of higher hydrocarbons

[0086] First, 150 g of the discharged solid was weighed, and put into a 2,500 ml sealed stainless steel container. Next, 1,500 ml of ethyl acetate (for high-performance liquid chromatography, FUJIFILM Wako Pure Chemical Corporation) was added to the discharged solid, and extraction was then performed for 3 hours in a warm bath at 40°C under hermetically stirring. After the extraction, the ethyl acetate solution and the extraction residue were separated with a stainless steel mesh having a mesh opening of 250 μm to obtain about 1,400 ml of an ethyl acetate solution. For dehydration treatment, 50 g of anhydrous sodium sulfate was added to the solution. The mixture was stirred for 5 minutes, then allowed to stand for a sufficient time, and filtered with filter paper to remove anhydrous sodium sulfate. The obtained ethyl acetate solution was placed into 500 ml-recovery flasks in an amount of 300 ml for each flask. The solvent was removed from each solution under reduced pressure with a rotary evaporator.

[0087] Next, 99% ethanol was added to each of the resulting dried solids so that the concentrations of the dried solids were 50% V/V, 10% V/V, 5% V/V, and 1% V/V, and the dried solids were dispersed and dissolved therein. The solutions were subjected to a dispersion treatment with an ultrasonic cleaner (SILENTSONIC UT-304, manufactured by Sharp Corporation) for 3 minutes, and it was confirmed that all the solids on the bottom surface of the recovery flask were dispersed. Then, the dispersions were respectively placed in four polypropylene centrifuge tubes (about 50 ml) in an amount of 40 ml for each centrifuge tube. The centrifuge tubes were allowed to stand for a whole day and night in a refrigerator for storing. Next, the resulting mixtures were centrifuged using a centrifuge (3700, manufactured by KUBOTA Corporation Co., Ltd.) at 3,000 rpm for 10 minutes. As a result, as shown in the table below, it was found that higher hydrocarbons could be efficiently removed by dilution at a high concentration of 10 wt% or more and precipitation with cooling.

[Table 3]

Dilution concentration	Dispersed state after dissolution	State after centrifugation
50%	Milky brown and slight precipitation	Clear brown liquid with precipitation layer
10%	Milky brown and slight precipitation	Clear brown liquid with precipitation layer
5%	Brown solution	Clear brown liquid with slight precipitation
1%	Brown solution	Clear brown liquid with trace amount of precipitation

[Example 4] Preparation of tobacco flavor preparation

[0088] First, 150 g of the discharged solid was weighed, and put into a 2,500 ml sealed stainless steel container. Next, 1,500 ml of ethyl acetate (for high-performance liquid chromatography, FUJIFILM Wako Pure Chemical Corporation) was added to the discharged solid, and extraction was then performed for 3 hours in a warm bath at 40°C under hermetically stirring. After the extraction, the ethyl acetate solution and the extraction residue were separated with a stainless steel mesh having a mesh opening of 250 μ m to obtain about 1,400 ml of an ethyl acetate solution. A 0.1% sulfuric acid aqueous solution was prepared in advance, and the aqueous solution and the previously obtained ethyl acetate solution were mixed at a solution ratio of 5 : 3 in a separatory funnel to obtain 800 ml of a mixed solution. Further, 50 g of salt was added to the mixed solution, and the mixture was sufficiently shaken in the separatory funnel to perform liquid-liquid extraction. At this time, alkaloids represented by nicotine migrated to the sulfuric acid aqueous solution in the lower layer, and hydrophobic active ingredients of tobacco leaves migrated to the ethyl acetate solution in the upper layer. After sufficient leaving, the ethyl acetate solution was taken out, and about 50 g of anhydrous sodium sulfate was added and stirred to perform a dehydration operation. The ethyl acetate solution was filtered with filter paper, and then ethyl acetate was removed under reduced pressure with a rotary evaporator (manufactured by Nihon Buchi) to obtain 7.7 g of a dried solid (yield: 5.2%). Next, 99% ethanol was added to the dried solid so that the content of the dried solid was 10%, and the mixture was stirred for dissolution at normal temperature. Further, the solution was allowed to stand in a sealed state in a refrigerator at about 5°C for a whole day and night, to generate an insoluble precipitate (corresponding to about 0.3 wt%). The precipitate was filtered off with filter paper to obtain a desired tobacco flavoring agent (clear brown alcohol preparation).

[0089] Shreds before expansion as a raw material of the discharged solid were evaluated using a component group having a target RI of 1,600 to 2,500 as an index. Specifically, a tobacco flavoring agent (alcohol preparation) was prepared in the same manner as in Example 4 except that shreds before expansion as a raw material were used, the tobacco flavoring agent was divided into small portions, and ethanol was removed therefrom under reduced pressure. Ethanol was added to the resulting dried solid so that the concentration of the dried solid was 4 wt%, and the dried solid was completely dissolved therein. Then, the resulting solution was subjected to GC/MS analysis under the conditions shown in Example 1. The results are shown in FIG. 6. The lower part shows the chromatogram of the extract of the shreds before expansion, and the upper part shows the chromatogram of the extract of the discharged solid (prepared in Example 4). In both of the case of using the shreds before expansion as a raw material and the case of using the discharged solid as a raw material, the component group having an RI of 1,600 to 2,500 was extracted. The upper chart had a larger peak area containing cembratrienediol (CBT) except for neophytadiene which is an unsaturated hydrocarbon. The result revealed that active aroma components were selectively concentrated in the discharged solid in the expansion process of the raw material. Further, from the viewpoint of the yield (discharged solid: 5.2%, shreds before expansion: 3.6%), it can be said that the extraction from the discharged solid is effective. The tobacco flavoring agent obtained in Example 4 had aroma peculiar to tobacco.

[Example 5] Confirmation of selective extraction of active aroma components

[0090] A small amount of the tobacco flavoring agent (alcohol preparation) obtained using ethyl acetate as an extraction solvent in Example 4 was separated, and ethanol was removed therefrom under reduced pressure. Ethanol was added to the resulting dried solid so that the concentration of the dried solid was 4 wt%, and the dried solid was completely dissolved therein. Then, the resulting solution was subjected to GC/MS analysis under the conditions shown in Experimental Example 1. When the peak area ratio was calculated based on the chromatogram in FIG. 7, the ratio of the component having an RI of less than 1,600 was 8.2%, the ratio of the component having an RI of 1,600 to 2,500 was 79.1%, and the ratio of the component having an RI of more than 2,500 was 12.7%. The results show that the component group having a desired RI of 1,600 to 2,500, that is, sesquiterpene and diterpene of the active aroma component could be selectively extracted.

[Example 6] Dissolution into propylene glycol

[0091] An alcohol preparation containing about 10 wt% of a tobacco extract (dried solid) was obtained in the same manner as in Example 4. Next, ethanol as a solvent was removed from the preparation with a rotary evaporator. Thereafter, propylene glycol was added to the resulting dried solid so that the concentration of the dried solid was 1 wt%, and the mixture was stirred for dissolution at normal temperature. The insoluble matter was then removed therefrom by filtration with a filter, to obtain a clear brown liquid (propylene glycol preparation). The flavoring agent obtained in this example had tobacco aroma equivalent to the flavoring agent prepared in Example 4.

[Example 7] Confirmation of effect with cigarette

[0092] The tobacco flavoring agent (alcohol preparation) obtained in Example 4 was added to shredded tobacco in amounts of 50 and 100 ppm with respect to the amount of the shredded tobacco. The obtained cased shreds were dried to obtain cased shreds that can be subjected to smoking evaluation. A cigarette was prepared using the cased shreds, and the cigarette was subjected to smoking evaluation. Smoking evaluation was performed by five well-trained panelists of an average age of 48 years. In the evaluation method, the intensity of the tobacco aroma was used as an index, and the evaluation was performed based on the following scores of: 1 point (no change) as no aroma; 2 points (strong); and 3 points (very strong). The test of the difference between the average values in the score was performed by two-sided test. The results demonstrated that the cased shreds exhibited better original tobacco aroma.

[Table 4]

Amount of 10% alcohol preparation added			
Subject	0 ppm	50 ppm	100 ppm
A	1	3	3
B	1	3	3
C	1	2	2
D	1	2	3
E	2	3	3
Average	1.2	2.6	2.8
Significant difference (p value)	-	0.002	0.0004

[Example 8] Confirmation of effect with non-combustion type tobacco

[0093] An amount of 2,000 ppm of the tobacco flavoring agent (alcohol preparation) obtained in Example 4 was added to tobacco base sheet shreds. The obtained flavored sheet shreds were dried to obtain flavored sheet shreds that can be subjected to smoking evaluation. A non-combustion heating type flavor inhalation article was prepared using the flavored sheet shreds. The article was heated from the outside using a heating apparatus set at 230 to 240°C in advance, and the same smoking evaluation as in Example 7 was performed. The results demonstrated that the flavored sheet shreds exhibited better original tobacco aroma than that of the unflavored sheet shreds.

[Example 9] Confirmation of effect with liquid-heated tobacco

[0094] An amount of 100 ppm of the tobacco flavoring agent (propylene glycol preparation) obtained in Example 6 was added to a base liquid containing 50% of glycerin and 50% of propylene glycol. A commercially available flavor inhalation article (Logic (trade mark)), which is a type of inhaling vapor obtained by vaporizing liquid, was filled with the tobacco flavoring agent. Smoking evaluation was performed using the article in the same manner as in Example 7. The results demonstrated that better original tobacco aroma was exhibited.

[Example 10] Preparation of tobacco flavor benzyl alcohol preparation

[0095] First, 150 g of the discharged solid was weighed, and put into a 2,500 ml sealed stainless steel container. Next, 1,500 ml of ethyl acetate (for high-performance liquid chromatography, FUJIFILM Wako Pure Chemical Corporation)

was added to the discharged solid, and extraction was then performed for 3 hours in a warm bath at 40°C under hermetically stirring. After the extraction, the ethyl acetate solution and the extraction residue were separated with a stainless steel mesh having a mesh opening of 250 μm to obtain about 1,400 ml of an ethyl acetate solution. A 0.1% sulfuric acid aqueous solution was prepared in advance, and the aqueous solution and the previously obtained ethyl acetate solution were mixed at a solution ratio of 5 : 3 in a separatory funnel to obtain 800 ml of a mixed solution. Further, 50 g of salt was added to the mixed solution, and the mixture was sufficiently shaken in the separatory funnel to perform liquid-liquid extraction. At this time, alkaloids represented by nicotine migrated to the sulfuric acid aqueous solution phase in the lower layer, and hydrophobic active ingredients of tobacco leaves migrated to the ethyl acetate solution phase in the upper layer. After sufficient leaving, the ethyl acetate solution was taken out, and about 50 g of anhydrous sodium sulfate was added and stirred to perform a dehydration operation. The ethyl acetate solution was filtered with filter paper, and then ethyl acetate was removed under reduced pressure with a rotary evaporator (manufactured by Nihon Buchi) to obtain 7.7 g of a dried solid (yield: 5.2%). Next, benzyl alcohol was added to the dried solid so that the content of the dried solid was 20 wt%, and the mixture was stirred for dissolution at normal temperature. The solution was allowed to stand in a sealed state in a refrigerator at about 5°C for a whole day and night, to generate an insoluble precipitate (corresponding to about 0.01 wt%). The precipitate was filtered off with filter paper to obtain a desired tobacco flavoring agent (clear brown benzyl alcohol preparation).

[0096] The benzyl alcohol preparation was divided into small portions, ethyl acetate was added thereto so that the concentration of the dried solid was 4 wt%, and the dried solid was completely dissolved therein. Then, the resulting solution was subjected to GC/MS analysis under the conditions shown in Experimental Example 1. FIG. 8 shows a chromatogram. As compared with the ethanol preparation shown in FIG. 7, the component group of the benzyl alcohol preparation was expanded to the component group having an RI of 1,600 to 3,500, and the content of saturated higher hydrocarbons was significantly increased. When the peak area ratio excluding benzyl alcohol was calculated, the ratio of the component having a retention index (RI) as an index of less than 1,600 was 1.3%, the ratio of the component having an RI of 1,600 to 2,500 was 87.5%, the ratio of the component having an RI of more than 2,500 and 3,500 or less was 8.8%, and the ratio of the component having an RI of more than 3,500 was 2.4%. The results show that saturated higher hydrocarbons, which are important for flavor, could be selectively extracted in addition to the desired component group having an RI of 1,600 to 3,500, that is, sesquiterpene and diterpene of the active aroma component.

[0097] A non-combustion heating type flavor inhalation article was prepared using the tobacco flavoring agent obtained in this example, in the same manner as in Example 8. By the same panelists as in Example 7, the article was heated from the outside using a heating apparatus with the heating temperature set to 270 to 280°C, and smoking evaluation was performed by the same panelists as in Example 7. In the evaluation method, the intensity of the tobacco aroma was used as an index, and the evaluation was performed based on the following scores of: 1 point (no change) as no aroma; 2 points (strong); and 3 points (very strong). The test of the difference between the average values in the score was performed by two-sided test. The results demonstrated that the article exhibited soft and smooth flavor characteristics in addition to the aroma peculiar to tobacco.

[Table 5]

Amount of 20% benzyl alcohol preparation added			
Subject	0 ppm	0.25%	0.75%
A	1	3	3
B	1	2	3
C	1	2	3
D	1	2	3
E	1	3	3
Average	1	2.4	3
Significant difference (p value)	-	0.004	0.001

[Example 11] Removal of insoluble component

[0098] First, 150 g of the discharged solid was weighed, and put into a 2,500 ml sealed stainless steel container. Next, 1,500 ml of ethyl acetate (for high-performance liquid chromatography, FUJIFILM Wako Pure Chemical Corporation) was added to the discharged solid, and extraction was then performed for 3 hours in a warm bath at 40°C under hermetically stirring. After the extraction, the ethyl acetate solution and the extraction residue were separated with a

stainless steel mesh having a mesh opening of 250 μm to obtain about 1,400 ml of an ethyl acetate solution. Further, 50 g of anhydrous sodium sulfate was added to the solution for dehydration treatment and the mixture was stirred for 5 minutes. Then, the mixture was allowed to stand for a sufficient time, and filtered with a filter paper, to remove anhydrous sodium sulfate. The obtained ethyl acetate solution was placed into 500 ml-recovery flasks in an amount of 300 ml for each flask. The solvent was removed from each solution under reduced pressure with a rotary evaporator.

[0099] Next, benzyl alcohol was added to each of the resulting dried solids so that the concentrations of the dried solids were 40% V/V, 20% V/V, 10% V/V, or 5% V/V, and the dried solids were dispersed and dissolved therein. At this time, the solutions were subjected to a dispersion treatment with an ultrasonic cleaner (SILENTSONIC UT-304, manufactured by Sharp Corporation) for 3 minutes, and it was confirmed that all the solids on the bottom surface of the recovery flask were dispersed. Then, the dispersions were respectively placed in four polypropylene centrifuge tubes (about 50 ml) at 40 ml for each centrifuge tube. The centrifuge tubes were allowed to stand for a whole day and night in a refrigerator for storing. Next, the mixtures were centrifuged using a centrifuge (3700, manufactured by KUBOTA Corporation Co., Ltd.) at 10,000 rpm for 1 hour. As a result, as shown in the table below, it was found that insoluble components could be efficiently removed by dilution at a concentration of 10 to 40 wt% and precipitation with cooling.

[Table 6]

Dilution concentration	Dispersed state after dissolution	State after centrifugation
40%	Viscous brown solution	Removal by centrifugation failed due to high viscosity
20%	Brown solution	Clear brown liquid with slight precipitation layer
10%	Brown solution	Clear brown liquid with slight precipitation
5%	Brown solution	No precipitation

[Example 12] Confirmation of effect with fine powder from raw material factory

[0100] A flue-cured variety raw material and a Burley variety raw material in the form of small lamina discarded from the treatment process of the tobacco leaf raw material and tobacco leaf fine powder collected by a dust collector were prepared.

[0101] Then, 150 g of each of the flue-cured variety raw material and the Burley variety raw material was weighed, and put into a 2,500 ml sealed stainless steel container. Next, 1,500 ml of ethyl acetate (for high-performance liquid chromatography, FUJIFILM Wako Pure Chemical Corporation) was added to each raw material, and extraction was then performed for 3 hours in a warm bath at 40°C under hermetically stirring. After the extraction, the ethyl acetate solution and the extraction residue were separated with a stainless steel mesh having a mesh opening of 250 μm to obtain about 1,400 ml of an ethyl acetate solution. Then, 300 ml of the obtained ethyl acetate solution and 500 ml of diluted sulfuric acid water adjusted to pH 2.0 were placed in a 1,000 ml separatory funnel, and the mixture was subjected to liquid-liquid extraction to recover an organic phase. The solvent was removed from the organic phase under reduced pressure with a rotary evaporator. Ethyl acetate was added to the concentrate so that the concentration of each dried solid was 4 wt%. The resulting solution was subjected to GC/MS analysis under the conditions shown in Experimental Example 1. FIGs. 17 and 18 show charts of the flue-cured variety raw material and the Burley variety raw material, respectively. As a result, results very similar to FIG. 8 were obtained. Further, each of the dried solids obtained above was dissolved in benzyl alcohol so that the content of the dried solid was 20 wt%, and a solid content was precipitated and removed in the same manner as in Example 11, to prepare each benzyl alcohol preparation. The obtained preparations and the preparation derived from the expansion process obtained in Example 10 were compared by sensory evaluation. Evaluation samples were prepared according to Example 10, and smoking evaluation was performed by the same panelists as in Example 7 with the heating temperature set at 270 to 280°C.

[Table 7]

Panelist	Discharged solid from expansion process	Flue-cured variety fine powder	Burley variety fine powder
A	3	3	3
B	3	2	2
C	3	3	3
D	2	3	3

(continued)

Panelist	Discharged solid from expansion process	Flue-cured variety fine powder	Burley variety fine powder
E	3	3	2
Average	2.8	2.8	2.6
Significant difference (p value)	-	2.306	2.306

[0102] The table shows that the preparations derived from the flue-cured variety raw material and the Burley variety raw material had very similar flavor characteristics.

REFERENCE SIGNS LIST

[0103]

- 10 Heating apparatus
- 11 Body
- 12 Heater
- 13 Metal tube
- 14 Battery unit
- 15 Control unit
- 16 Recess
- 17 Vent hole
- 20 Non-combustion heating type flavor inhalation article
- 20A Tobacco rod part
- 20B Cooling part
- 20C Filter part
- 21 Tobacco filler
- 22 Cigarette paper
- 23 Paper tube
- 24 Perforation
- 25 First segment
- 25a First filling layer
- 25b Inner plug wrapper
- 26 Second segment
- 26a Second filling layer
- 26b Inner plug wrapper
- 27 Outer plug wrapper
- 28 Lining paper
- 30 Non-combustion non-heating type flavor inhalation article
- 30D Power supply unit
- 30E Cartridge
- 30F Tobacco capsule
- u Non-inhalation end
- d Inhalation end
- 110 Battery
- 200 Aerosol source
- 210 Reservoir
- 220 Atomizer
- 230 Flow path forming body
- 240 Outer frame 240
- 250 End cap

200X First flow path
 300 Flavor source
 310 Container
 320 Mesh body
 5 330 Nonwoven fabric
 340 Cap

Claims

1. A method for producing a tobacco extract containing a tobacco terpene, the method comprising:
 - a process 1 that includes preparing a raw material derived from tobacco;
 - a process 2 that includes subjecting the raw material to solid-liquid extraction with an aprotic medium-polar solvent containing a hetero element;
 - a process 3 that includes recovering an organic phase from the process;
 - a process 4 that includes adding a protic polar solvent or an aprotic medium-polar solvent to an extract obtained by removing the solvent from the organic phase, to precipitate or disperse a solid content; and
 - a process 5 that includes removing the solid.
2. The production method according to claim 1, wherein a boiling point of the aprotic medium-polar solvent used in the process 2 is 80°C or lower.
3. The production method according to claims 1 and 2, wherein the aprotic medium-polar solvent is selected from the group consisting of ethyl acetate, butyl butyrate, ethyl butyrate, dichloromethane, chloroform, and a combination thereof.
4. The production method according to any one of claims 1 to 3, wherein the aprotic medium-polar solvent is ethyl acetate.
5. The production method according to any one of claims 1 to 4, wherein the process 2 further includes subjecting an organic phase obtained by solid-liquid extraction to extraction with water or an acid aqueous solution, and acid used for the acid aqueous solution is an inorganic acid or an organic acid.
6. The method for producing a tobacco extract according to any one of claims 1 to 5, wherein the acid is selected from the group consisting of sulfuric acid, citric acid, oxalic acid, and a combination thereof.
7. The production method according to any one of claims 1 to 6, wherein the process 2 further includes subjecting an organic phase obtained by solid-liquid extraction to extraction with water or an acid aqueous solution, and a pH of the acid aqueous solution is 4 or less.
8. The production method according to any one of claims 1 to 7, wherein the protic polar solvent used in the process 4 is ethanol.
9. The production method according to any one of claims 1 to 7, wherein the aprotic medium-polar solvent used in the process 4 is benzyl alcohol.
10. The production method according to any one of claims 1 to 9, wherein the process 4 includes adding the solvent to the extract to form a solution once, and then precipitating a solid content.
11. The production method according to any one of claims 1 to 9, wherein the process 4 includes adding the solvent to the extract, to obtain a dispersion in which a solid content is dispersed.
12. The production method according to claim 11, further comprising promoting precipitation of the solid content.
13. The production method according to any one of claims 1 to 12, wherein a concentration of the extract in the solution or the dispersion prepared in the process 4 is 5 to 20 wt%.

14. A tobacco extract obtained by the method according to claims 1 to 13.

15. A tobacco flavoring agent comprising the tobacco extract according to claim 14.

5 **16.** A tobacco material comprising the tobacco flavoring agent according to claim 15.

17. The tobacco material according to claim 16, wherein the tobacco material is a tobacco sheet or a shredded tobacco.

10 **18.** A tobacco rod part comprising the tobacco material according to claim 16 or 17.

19. A tobacco flavor inhalation article comprising the tobacco rod part according to claim 18.

15 **20.** A non-combustion heating type tobacco flavor inhalation article or a non-combustion non-heating type tobacco flavor inhalation article comprising the tobacco rod part according to claim 18.

21. A smokeless tobacco comprising the tobacco extract according to claim 14.

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FIG. 1

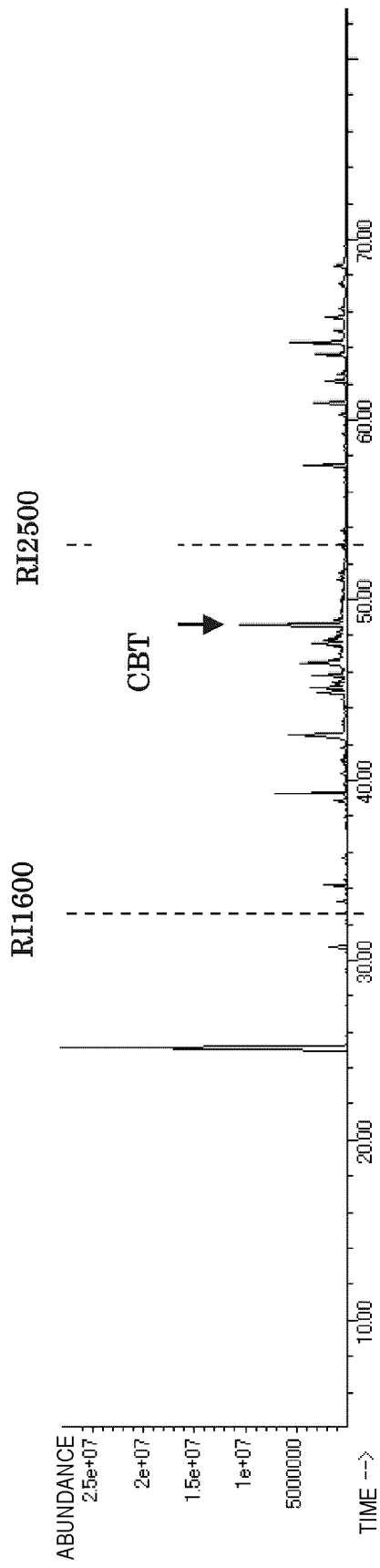


FIG. 2

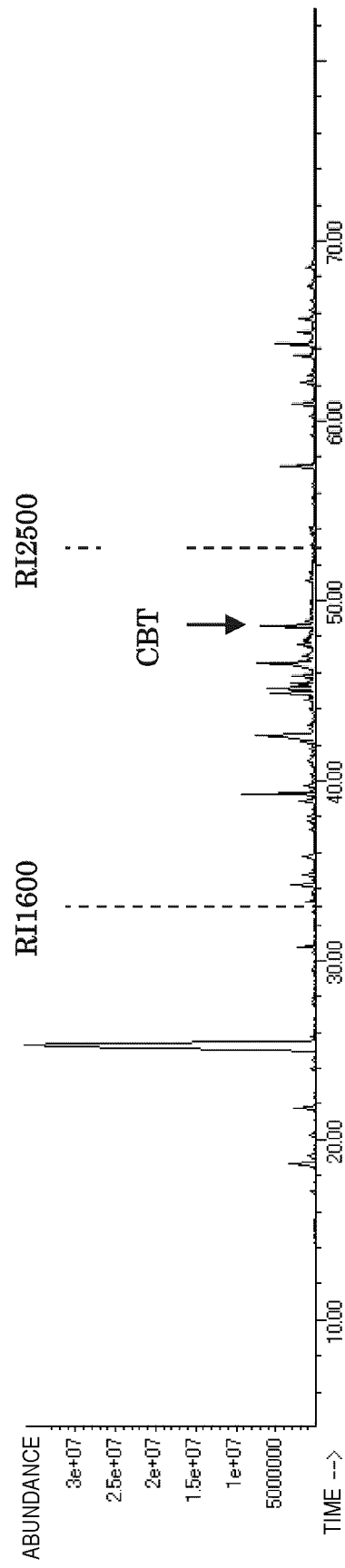


FIG. 3

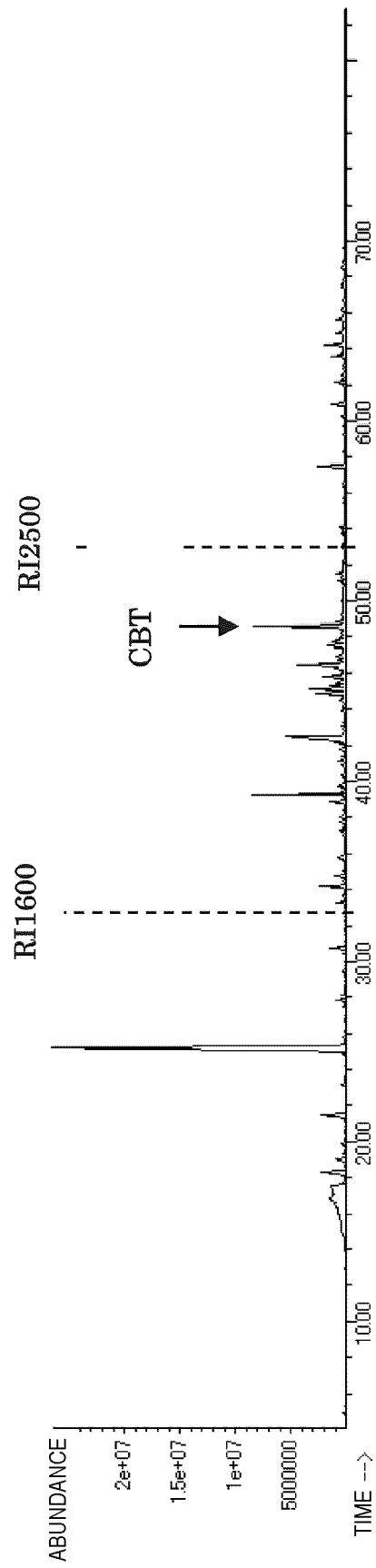


FIG. 4

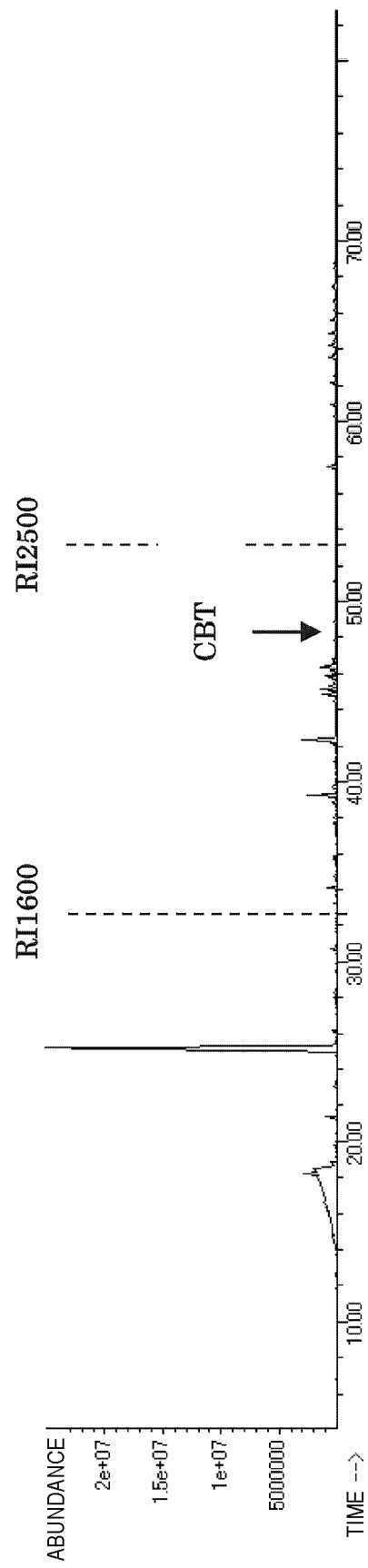


FIG. 5

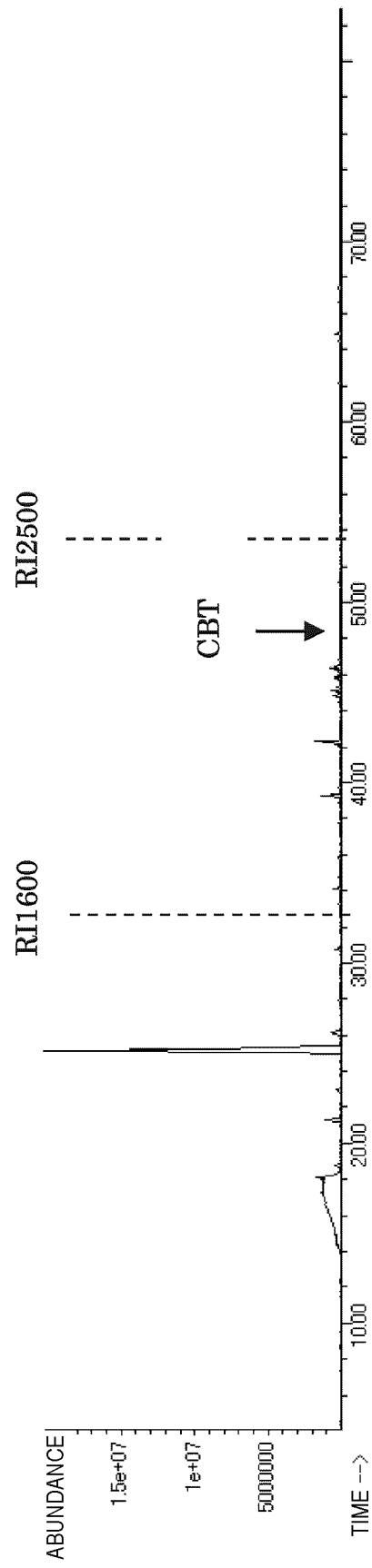


FIG. 6

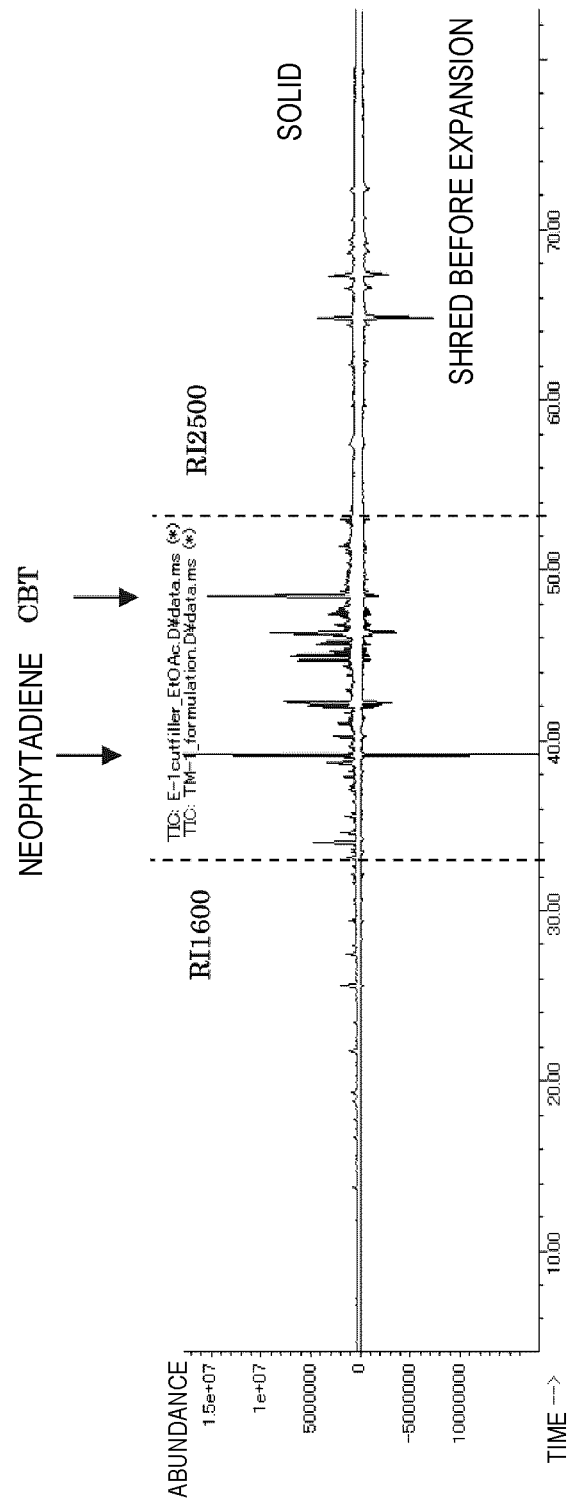


FIG. 7

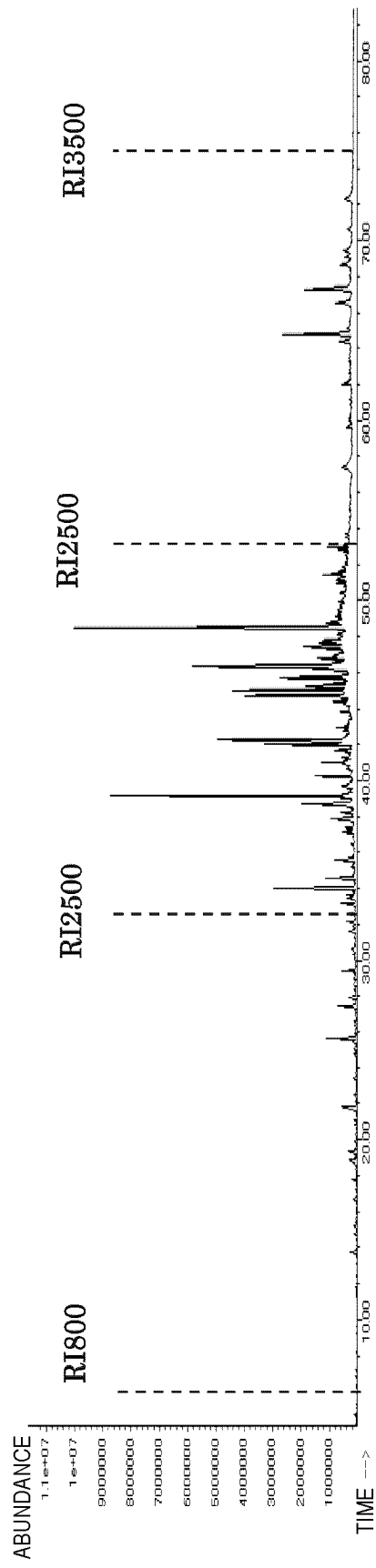


FIG. 8

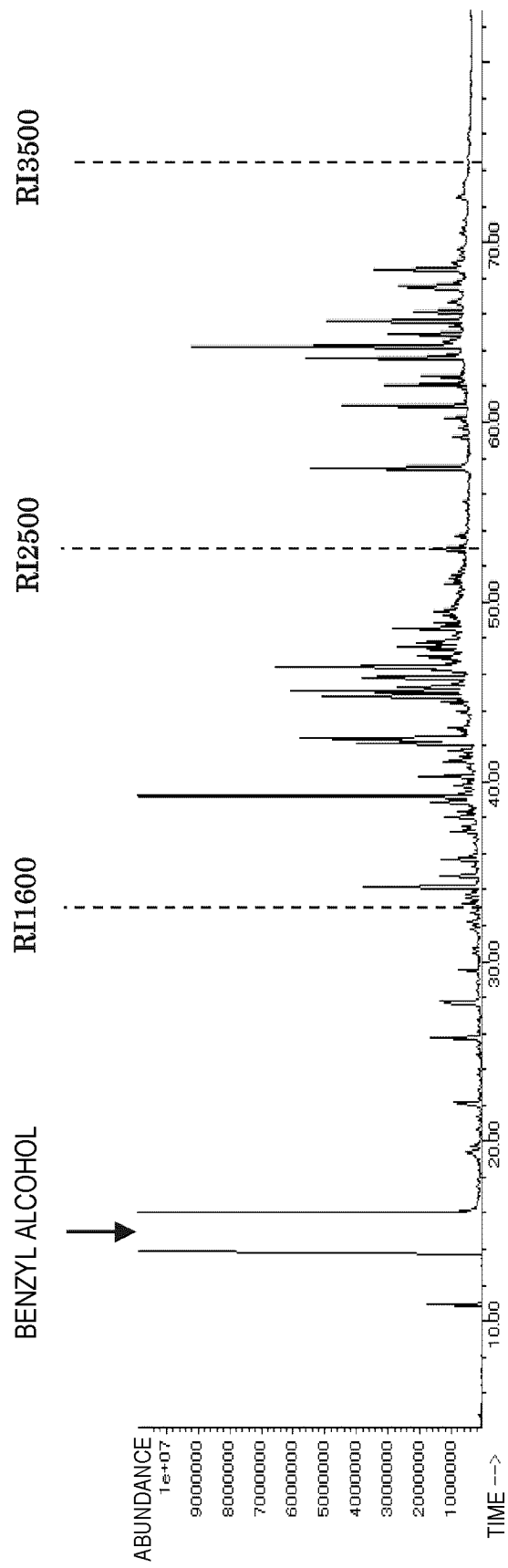


FIG. 9

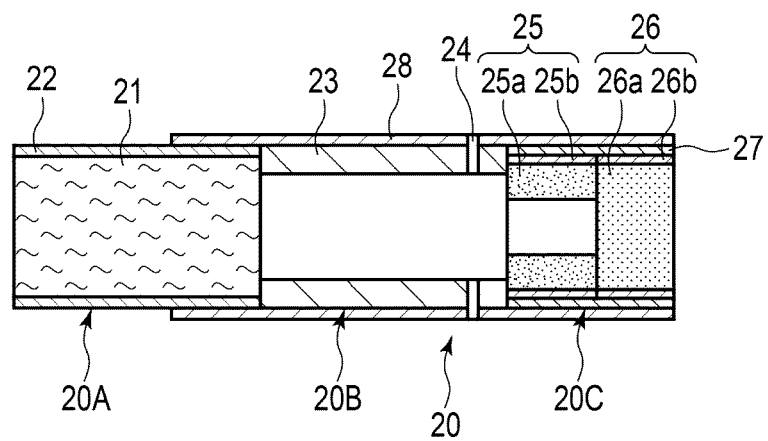


FIG. 10

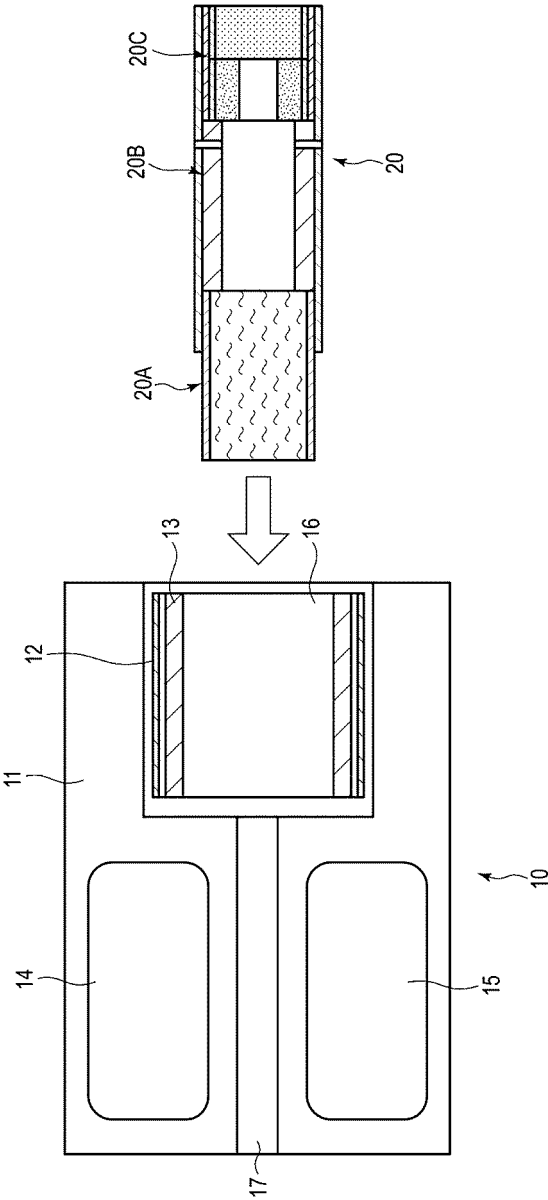


FIG. 11

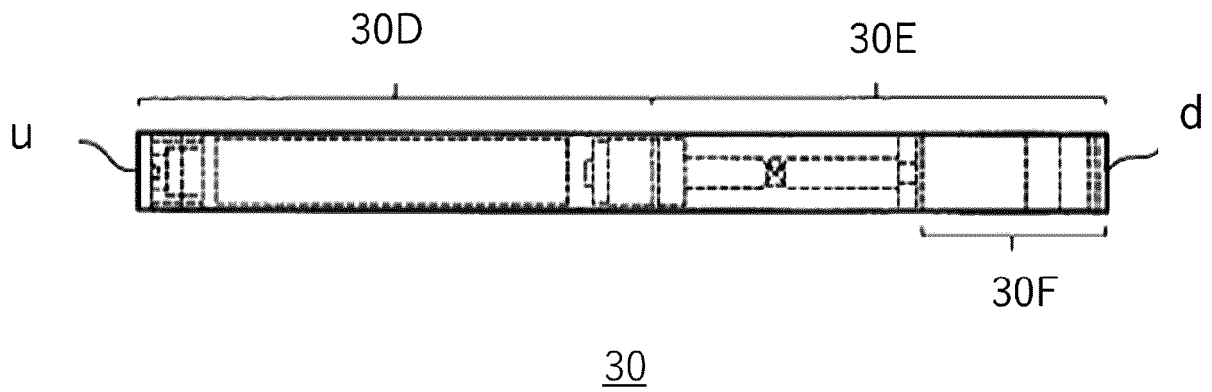


FIG. 12

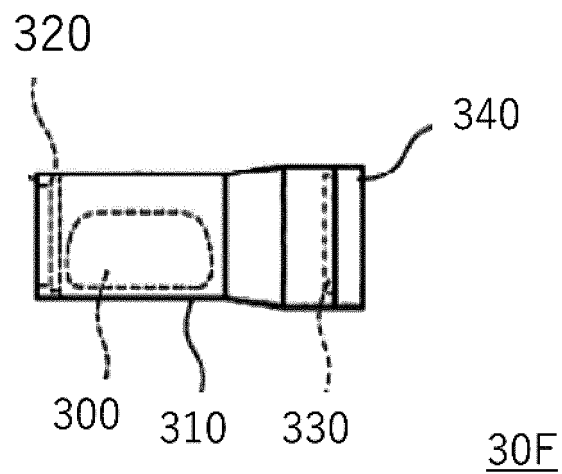


FIG. 13

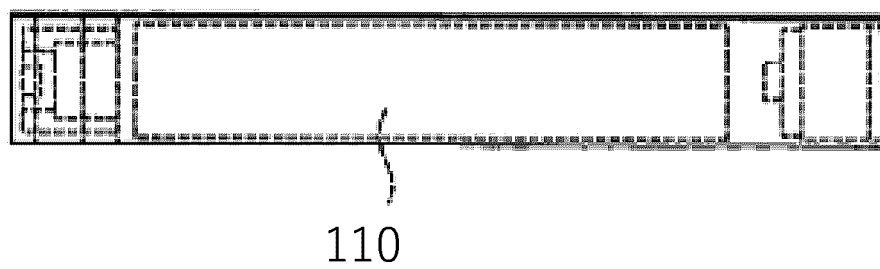


FIG. 14

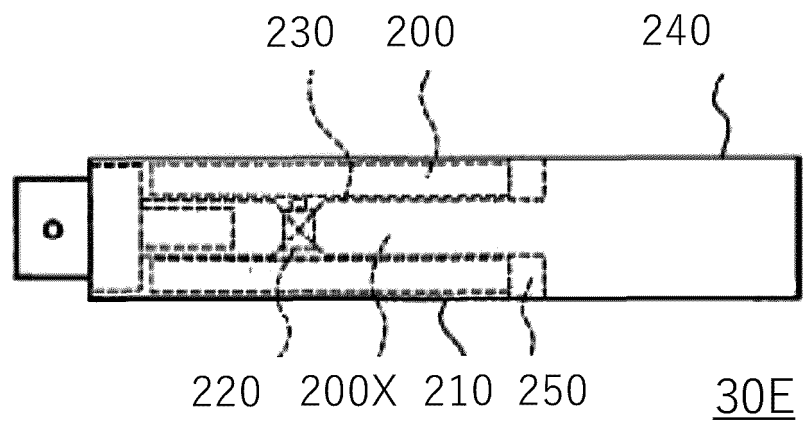


FIG. 15

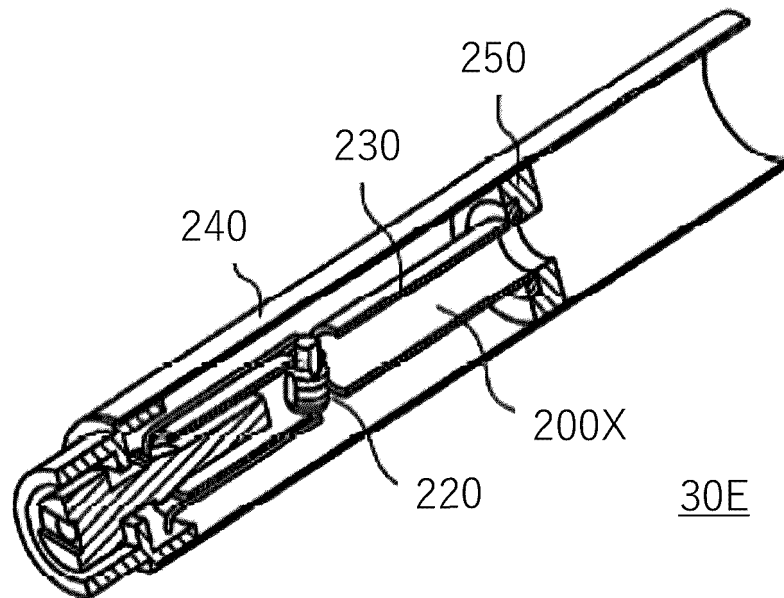


FIG. 16

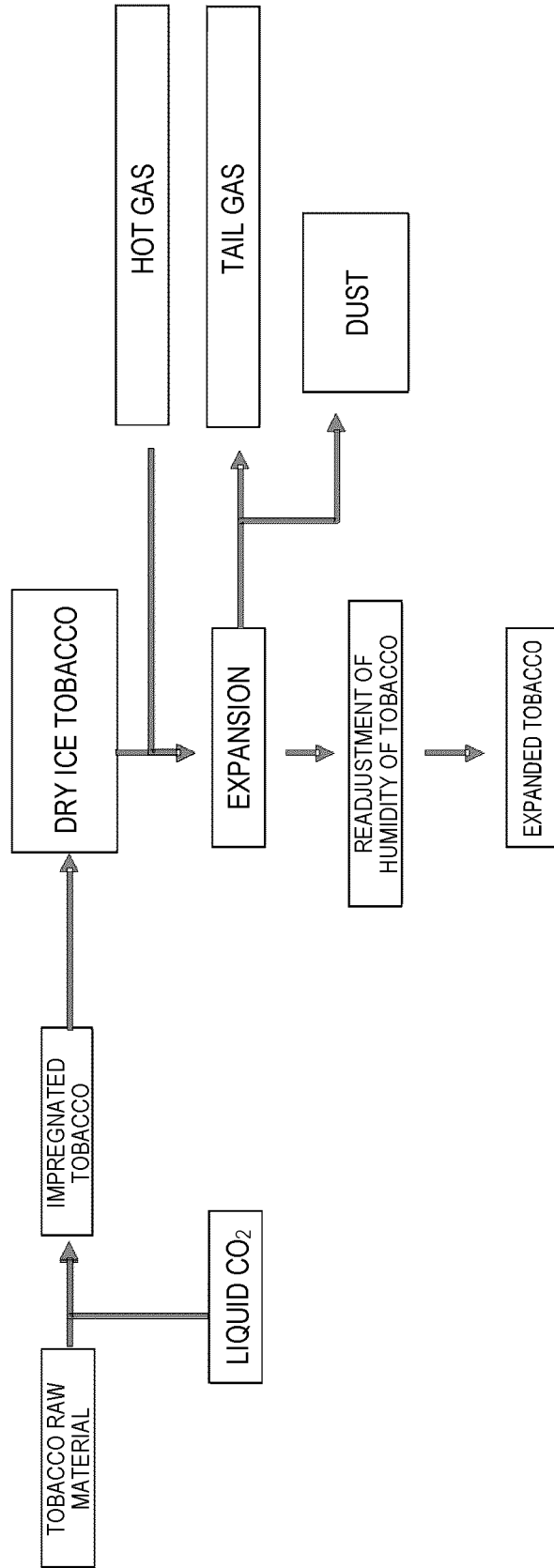


FIG. 17

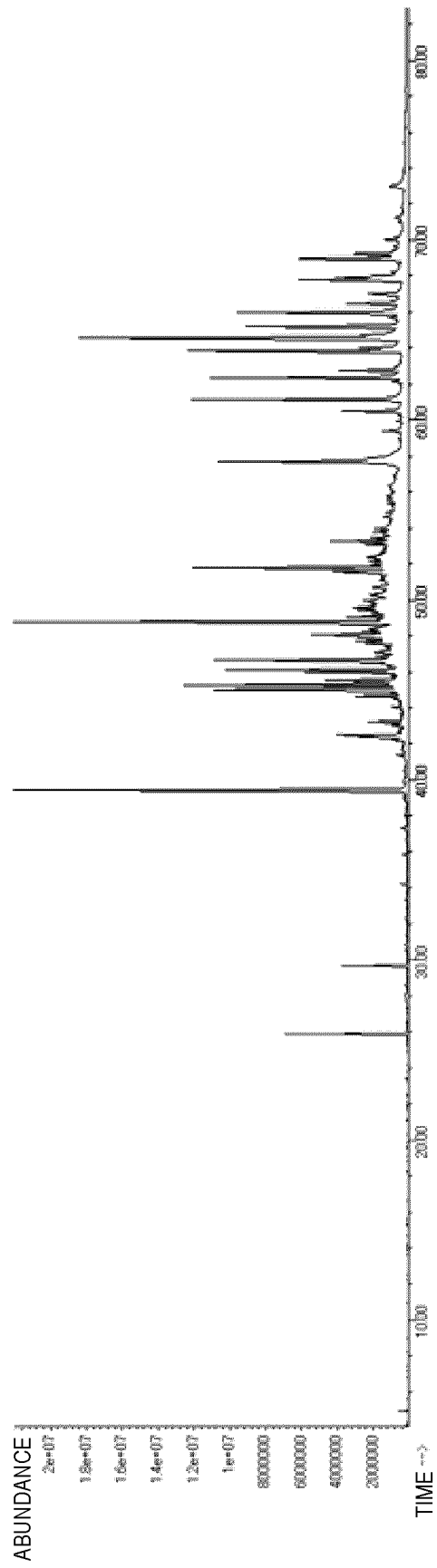
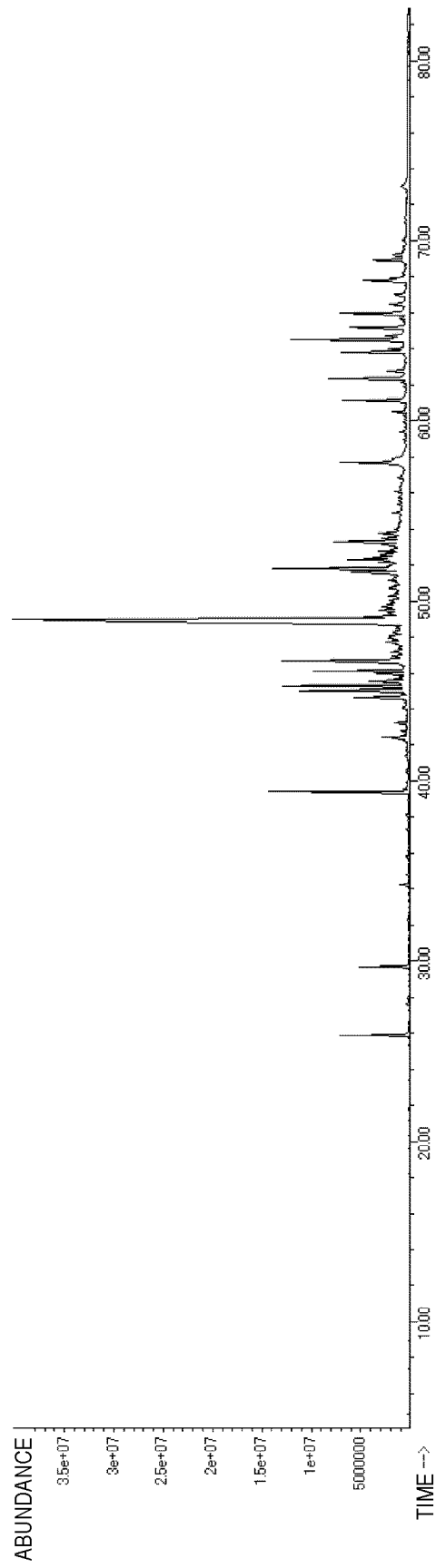


FIG. 18



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2021/040833

A. CLASSIFICATION OF SUBJECT MATTER

A24B 15/167(2020.01)i; **A24B 15/26**(2006.01)i; **C11B 9/00**(2006.01)i; **A24B 13/00**(2006.01)i
FI: A24B15/26; A24B15/167; A24B13/00; C11B9/00 B

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

A24B15/167; A24B15/26; C11B9/00; A24B13/00

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

Published examined utility model applications of Japan 1922-1996
Published unexamined utility model applications of Japan 1971-2021
Registered utility model specifications of Japan 1996-2021
Published registered utility model applications of Japan 1994-2021

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Y	JP 2004-258012 A (JAPAN SCIENCE & TECHNOLOGY AGENCY) 16 September 2004 (2004-09-16) paragraph [0032]	1-21
Y	JP 2009-167176 A (KAO CORP) 30 July 2009 (2009-07-30) paragraph [0015]	1-21
Y	JP 2003-252876 A (NITTO DENKO CORP) 10 September 2003 (2003-09-10) paragraph [0019]	4-21

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

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

Date of the actual completion of the international search

22 December 2021

Date of mailing of the international search report

11 January 2022

Name and mailing address of the ISA/JP

Japan Patent Office (ISA/JP)
3-4-3 Kasumigaseki, Chiyoda-ku, Tokyo 100-8915
Japan

Authorized officer

Telephone No.

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/JP2021/040833

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JP 2009-167176 A	30 July 2009	(Family: none)	
JP 2003-252876 A	10 September 2003	(Family: none)	

Form PCT/ISA/210 (patent family annex) (January 2015)

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