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(54) TOBACCO MATERIAL PRODUCTION METHOD

(57) A method of producing a tobacco material, including a step of impregnating a tobacco raw material with a medium in the state selected from the group consisting of gas, liquid, and supercritical states or thus a step of obtaining a tobacco material having a surface migration rate X of more than 1 defined as surface migration rate X = S/P, where S is a GC peak area for a flavor component present on the surface of a tobacco raw material after impregnation, and P is a GC peak area for the flavor component present on the surface of the tobacco raw material before impregnation.

Processed by Luminess, 75001 PARIS (FR)

Description

TECHNICAL FIELD

[0001] The present invention relates to a production method for a tobacco material.

BACKGROUND ART

[0002] Aroma constituents of tobacco leaves have been studied for various tobacco raw materials, such as flue-cured, burley, oriental, and Maryland tobacco, and characteristic constituents have been reported for the respective varieties. More than 4000 such constituents have been reported so far (Non-Patent Literature (NPL) 1). Constituents that contribute to characteristic tobacco leaf aromas are broadly divided into two groups in terms of localization inside or outside the cells of tobacco leaves. The first group includes constituents that are present in the cell tissues of tobacco leaves. These are aromas of degradation products that are primarily derived from pigments and are formed in the drying process or in the maturation period of tobacco leaves, in other words, ionones, megastigmatrienone, phytol, other pigment degradation products, monoterpenes, and phenols. All of these constituents are accumulated inside the cell tissues of tobacco leaves through common metabolic pathways associated with the generation of aroma constituents, such as the shikimate pathway, the mevalonate pathway, and pigment degradation pathways.

[0003] The second group includes constituents present on the surfaces of tobacco leaves. These are commonly referred to as leaf surface resin and comprise, for example, relatively high-molecular organic acids, hydrocarbons, and glycosides that are produced and secreted from trichomes and so forth to protect tissues from pests or the growing environment. Such aroma constituents are characteristically sesquiterpenes, diterpenes, and terpene glycosides thereof.

[0004] There have been proposed various methods of extracting aroma constituents from tobacco leaves and so forth. By exploiting the feature of a target constituent, an extraction method using liquid carbon dioxide or supercritical carbon dioxide has been proposed (Patent Literature (PTL) 1 and 2, for example). As a raw material for combustible cigarettes, a raw material of tobacco leaves expanded with liquid carbon dioxide has been investigated, where the expansion process is extremely similar to the extraction method using carbon dioxide. For this reason, a method to which part of the expansion process is applied has been proposed (PTL 3 and 4, for example). PTL 3 discloses an apparatus for producing a flavor for an expanded tobacco raw material. The apparatus includes: an extraction vessel for bringing a tobacco raw material into contact with supercritical carbon dioxide to dissolve tobacco components in carbon dioxide; a separation vessel, which is connected to the extraction vessel, for separating and recovering fat-soluble parts from the tobacco components dissolved in carbon dioxide; a circulation path for circulating, between the extraction vessel and an absorption vessel in which purified water is stored, supercritical carbon dioxide after the separating and recovering while purifying with a purification layer of activated carbon and allowing the purified water to absorb water-soluble parts of the tobacco components; and a collecting vessel for collecting, as absorption water from the absorption vessel, the purified water that has absorbed water-soluble parts of the tobacco components. The fat-soluble parts of the tobacco components and the absorption water are used for producing flavors.

CITATION LIST

PATENT LITERATURE

[0005]

PTL 1: WO 2007/029264
PTL 2: UK Patent No. 2173985
PTL 3: WO 2007/119790

NON PATENT LITERATURE

[0006]

NPL 1: Leffingwell et al., Leffingwell Reports, Vol. 1 (No. 2), February 2001
NPL 2: Ha Si et al., Discoloration and Strength Reduction of Plant Fibers by Heat Degradation, Sen'I Gakkaishi, pp. 89-95, Vol. 70, No. 5, 2014,
NPL 3: J. Sui et al., Formation of α- and β-Cembratriene-Diols in Tobacco (Nicotiana tabacum L.) Is Regulated by Jasmonate-Signaling Components via Manipulating Multiple Cembranoid Synthetic Genes, Molecules, 23(10): 2511, 2018

NPL 4: W. S. Schlotzhauer et al., Characterization of thermolysis products of cuticular wax compounds of green tobacco leaf, Journal of Analytical and Applied Pyrolysis, Volume 17, Issue 1, December 1989, Pages 25-35

SUMMARY OF INVENTION

TECHNICAL PROBLEM

[0007] In the case of combustible cigarettes, the above-mentioned aroma constituents are distilled and thermally decomposed during combustion/decomposition of cell structures. In other words, aroma constituents of leaf tobacco, regardless of being localized on the surfaces or inside cells of tobacco leaves as described above, readily migrate into mainstream smoke. It is thus considered the effect of localization is extremely small.

[0008] Meanwhile, in the case of non-combustion cigarettes, since the movement inside cells is restricted by shields of cell walls and other structures, constituents that are localized on the surfaces, primarily such as leaf surface resin, are preferentially volatilized as compared with conventional combustible cigarettes. It is thus considered that a heating temperature is important in non-combustion cigarettes for releasing aroma constituents present inside cells and that heating to 200°C or higher, which is the heat denaturation temperature of cell wall constituents, is required (NPL 2). For example, since ionones and so forth, which are degradation products of carotenes contained in plastids (NPL 1), are present inside cells, the release thereof is suppressed at a low temperature. Meanwhile, it is anticipated that the controlled delivery is not easy since these constituents are immediately volatilized at the heat denaturation temperature simultaneously with the heat degradation of cell wall constituents. In contrast, cembratrienediol (CBT) (NPL 3), which is a leaf surface resin component, has a low vapor pressure and is thus expected to be delivered in response to the amount of heat applied as contrasted to the above-mentioned aroma constituents present inside cells. In other words, since the release of aroma constituents is restricted in non-combustion cigarettes depending on places in which the aroma constituents are present, it is not easy to release aroma constituents as in combustible cigarettes. In view of this, the object of the present invention is to provide a tobacco material that readily releases aroma constituents.

SOLUTION TO PROBLEM

[0009] The present inventors focused on the facts that aroma constituents present inside the cells of leaf tobacco move from the inside to the outside of the cells in expanding processing of tobacco leaves when carbon dioxide is removed through vaporization after impregnation with carbon dioxide gas and that such aroma constituents could potentially be mixed, in cell surface layers, with less volatile substances, such as leaf surface resin, which are originally present outside cells and thus dissolved or retained therein. On the basis of these facts, the present invention has been completed. In other words, the object is attained by the present invention below.

Embodiment 1

[0010] A method of producing a tobacco material, including a step of impregnating a tobacco raw material with a medium in the state selected from the group consisting of gas, liquid, and supercritical states to obtain a tobacco material having a surface migration rate X defined below of more than 1.

$$\text{Surface migration rate } X = S/P$$

S: a GC peak area for a flavor component present on the surface of a tobacco raw material after impregnation
P: a GC peak area for the flavor component present on the surface of the tobacco raw material before impregnation
Embodiment 2

[0011] The method according to Embodiment 1, where the medium is carbon dioxide, water, or an organic solvent.
Embodiment 3
[0012] The method according to Embodiment 1 or 2, where the medium is liquid carbon dioxide or supercritical carbon dioxide.

Embodiment 4

[0013] The method according to any of Embodiments 1 to 3, where the step of impregnating is conducted under a pressure of 2 MPa or more.

Embodiment 5

[0014] The method according to any of Embodiments 1 to 4, where the step of impregnating is conducted for 10 minutes or more.

Embodiment 6

[0015] The method according to any of Embodiments 1 to 5, where S and P are determined under the following conditions.

[Conditions]

[0016]

1) Five grams of a tobacco raw material after impregnation is weighed.
2) Into a container, 100 mL of an aprotic solvent and then the weighed sample are fed.
3) Subsequently, the container is shaken for 30 seconds for extraction.
4) The resulting extract is filtered to obtain a filtrate.
5) The extract obtained in 4) is filtered preferably by using a filter paper.
6) The filtered extract is added with a desiccant and dehydrated overnight.
7) The desiccant is filtered out preferably by using a filter paper, and the resulting filtrate is concentrated under reduced pressure to obtain a dry solid. The dry solid is dissolved in the aprotic solvent to prepare a 4% solution and analyzed by GC/MS to obtain the S value.
8) The tobacco raw material before impregnation is subjected to the procedure of 1) to 7) to obtain the P value.

Embodiment 7

[0017] The method according to any of Embodiments 1 to 6, where the tobacco raw material is conditioned or unconditioned tobacco leaves, laminae, stems, stalks, or shreds.

Embodiment 8

[0018] The method according to any of Embodiments 1 to 7, further including a step of pulverizing the tobacco raw material after the impregnating.

Embodiment 9

[0019] The method according to any of Embodiments 1 to 8, where the tobacco raw material is subjected to, after the impregnating, pulverization without separating from or after separating from the medium.

Embodiment 10

[0020] The method according to Embodiment 9, where the pulverization is freeze grinding.

Embodiment 11

[0021] A tobacco material obtained by the method of Embodiments 1 to 10.

Embodiment 12

[0022] The tobacco material according to Embodiment 11, having a D90 of 30 μm or less.

ADVANTAGEOUS EFFECTS OF INVENTION

[0023] According to the present invention, it is possible to provide a tobacco material that readily releases aroma constituents.

BRIEF DESCRIPTION OF DRAWINGS

[0024]

Fig. 1 illustrates an embodiment of a heat-not-burn tobacco flavor inhaler article.
Fig. 2 illustrates an embodiment of a heat-not-burn tobacco flavor inhaler system.
Fig. 3 shows gas chromatograms.

DESCRIPTION OF EMBODIMENTS

[0025] Hereinafter, the present invention will be described in detail. In the present invention, the expression of "X to Y" includes the lower and the upper limits of X and Y.

1. Production Method

[0026] A production method of the present invention includes a step of impregnating a tobacco raw material with a medium in the state selected from the group consisting of gas, liquid, and supercritical states to obtain a material having a surface migration rate X defined below of more than 1.

$$\text{Surface migration rate } X = S/P$$

S: a GC peak area for an aroma constituent present on the surface of a tobacco raw material after impregnation
P: a GC peak area for the aroma constituent present on the surface of the tobacco raw material before impregnation

(1) Medium

[0027] A medium is not limited provided that an aroma constituent can be extracted from a tobacco raw material, and carbon dioxide, water, or an organic solvent may be used therefor. Exemplary organic solvents include aprotic solvents, such as pentane, hexane, benzene, diethyl ether, and ethyl acetate. Among these, carbon dioxide is preferably used as a medium since expanding processing of tobacco leaves can be utilized.

[0028] Such a medium may be in the gas, liquid, or supercritical state but is preferably in the liquid or supercritical state in view of handling properties. In particular, liquid or supercritical carbon dioxide is preferably used as a medium since expanding processing of tobacco leaves can be utilized as mentioned above.

(2) Tobacco Raw Materials

[0029] A tobacco raw material is not limited provided that the raw material is derived from tobacco plants but is preferably tobacco leaves, laminae, stems, stalks, or shreds in view of easy availability and so forth. The term "tobacco leaves" is a generic term for harvested tobacco leaves before aging. Such aging encompasses curing as an embodiment. Laminae are tobacco leaves from which stems have been removed, and hence, the removed are stems. Stalks are those remaining on the field after harvesting tobacco. Shreds are aged tobacco leaves, stems, and so forth that have been shredded into a predetermined size. In particular, stems that have been shredded into a predetermined size are also referred to as stem shreds. These materials may be conditioned. Conditioning is performed to make the water content constant. For example, laminae can be conditioned by storing at 22°C and 60% for 24 hours or more. The upper limit of the time for storing is not limited but is preferably 30 hours or less. When the water content of a raw material is high, a medium readily diffuses inside cells due to swelling of the raw material, thereby facilitating the movement of an aroma constituent present inside cells outside. Meanwhile, when the water content of a raw material is high, drying is required after processing. In view of this, the water content of a raw material after conditioning is preferably 5 to 40 weight% and more preferably 10 to 30 weight%.

(3) Impregnation Step

[0030] Impregnation indicates allowing the medium to permeate a tobacco raw material. The medium may permeate at least part of a tobacco raw material but preferably the entire tobacco raw material. The pressure and temperature during impregnation are adjusted for a desirable state of a medium. For example, when liquid carbon dioxide is used, the ambient pressure (pressure inside a vessel, for example) of a tobacco raw material is preferably 2 MPa or more, more preferably 3 MPa or more, and further preferably 5 MPa or more (all as gauge pressure). The upper limit for the pressure is not limited but may be set to less than 100 MPa, for example. The temperature in this case may be set to -10°C to 35°C. When supercritical carbon dioxide is used, the pressure is preferably 100 MPa or more and more preferably 200 MPa or more (all as gauge pressure). The upper limit for the pressure is not limited but may be set to 300 MPa or less, for example. The ambient temperature (the material temperature or the temperature inside a vessel, for example) of a tobacco raw material in this case may be set to higher than 35°C to 70°C.

[0031] Through the impregnation step, an aroma constituent present inside the cells of a tobacco raw material moves outside the cells and further to the surface of the tobacco raw material. The reason is not limited but is presumably because a medium that has permeated into a tobacco raw material comes into contact with cells to generate a concentration gradient of an aroma constituent between the inside and the outside of the cells, thereby allowing the aroma constituent present inside the tobacco raw material to move to the medium side. Moreover, in the case of using carbon dioxide, carbon dioxide that has permeated into a tobacco raw material additionally cools water within tobacco leaves to form ice inside cells, thereby partially destroying organs that store aromas, such as oil glands or vacuoles. Consequently, an aroma constituent present inside the tobacco raw material further readily moves to the medium side. For smoother such movement, a medium preferably permeates the inside of the tobacco raw material. However, when the aroma constituent is completely dissolved or dispersed in the medium, it is difficult for the aroma constituent to remain on the surface of the tobacco raw material when the medium is removed. For this reason, it is possible in the present invention to allow an aroma constituent that has been present inside cells to remain on the surface of a tobacco raw material by the combination of efficient permeation of a solvent into a raw material cells to dissolve a desirable aroma constituent in the solvent and the removal of the solvent under milder conditions as possible.

[0032] Through impregnation, a surface migration rate X defined below is adjusted to more than 1.

$$\text{Surface migration rate } X = S/P$$

S: a GC peak area for an aroma constituent present on the surface of a tobacco raw material after impregnation
P: a GC peak area for the aroma constituent present on the surface of the tobacco raw material before impregnation

[0033] The surface migration rate X is preferably 1.2 or more, more preferably 1.5 or more, and further preferably 2 or more.

[0034] S is obtained by collecting an aroma constituent present on the surface of the processed tobacco raw material and analyzing by gas chromatography (GC). Specifically, the tobacco raw material is brought into contact with an aprotic solvent, such as ethyl acetate, to extract an aroma constituent present on the surface. Subsequently, the aprotic solvent is removed, and the resulting extract is weighed. Later, a peak area for the aroma constituent is obtained by GC. P is obtained in the same manner as for S by using the tobacco raw material before the processing.

[0035] S and P are more preferably defined as follows.

S: a GC peak area for an aroma constituent extracted under the conditions below from a tobacco raw material after impregnation
P: a GC peak area for the aroma constituent extracted under the conditions below from the tobacco raw material before impregnation

[Conditions]

[0036]

1) Five grams of a tobacco raw material after impregnation is weighed.
2) Into a container (preferably, a 200 mL beaker), 100 mL of an aprotic solvent and then the weighed sample are fed.
3) Subsequently, the container is shaken for 30 seconds for extraction (so as to wash the surface of the raw material).
4) The resulting extract is filtered to obtain a filtrate (the filtrate is preferably received by a 200 mL Erlenmeyer flask).
5) The extract obtained in 4) is filtered. A filter paper is preferably used on this occasion.
6) The filtered extract is added with a desiccant and dehydrated overnight.
7) The desiccant is filtered out, and the resulting filtrate is concentrated under reduced pressure to obtain a dry solid. A filter paper is preferably used on this occasion. The dry solid is dissolved in the aprotic solvent to prepare a 4% solution and analyzed by GC/MS to obtain the S value.
8) The tobacco raw material before impregnation is subjected to the procedure of 1) to 7) to obtain the P value.

[0037] In a more preferable embodiment, extraction is conducted at two stages, specifically as follows.

[Conditions]

[0038]

1) Five grams of a tobacco raw material after impregnation is weighed.
2) Into a container (preferably, a 200 mL beaker), 100 mL of an aprotic solvent and then half the amount of the weighed sample are fed.
3) Subsequently, the container is shaken lightly for 30 seconds for extraction (so as to wash the surface of the raw material).
4-1) The resulting extract is filtered, and the filtrate is received by another container (preferably, a 200 mL Erlenmeyer flask).
4-2) The resulting filtrate is added with the remaining raw material and subjected to extraction for 30 seconds in the same manner as 3).
5) The extract obtained in 4) is filtered. A filter paper is preferably used on this occasion.
6) The filtered extract is added with a desiccant and dehydrated overnight.
7) Anhydrous sodium sulfate is filtered out, and the resulting filtrate is concentrated under reduced pressure to obtain a dry solid. A filter paper is preferably used on this occasion. The dry solid is dissolved in the aprotic solvent to prepare a 4% solution and analyzed by GC/MS to obtain the S value.
8) The tobacco raw material before impregnation is subjected to the procedure of 1) to 7) to obtain the P value.

[0039] Exemplary aprotic solvents include ester solvents, such as ethyl acetate. The amount of desiccant may be set to about 1 to 5 g and more preferably 2 to 4 g. Anhydrous inorganic salts, such as anhydrous sodium sulfate, may be used as desiccants.

[0040] Exemplary aroma constituents for which the above-described surface migration rate X is determined include: terpene aroma constituents from the mevalonate pathway, such as monoterpenes and sesquiterpenes: e.g. limonene, geraniol, and farnesol; phenol aroma constituents derived from lignin or shikimate pathway such as eugenol, anethole, and vanillin; fatty acids or linear unsaturated hydrocarbons, such as palmitic acid, myristic acid, oleic acid, and solanesol; amino acid-derived aroma constituents, such as indole and skatole; alkaloids, such as nicotine unique to tobacco; and pigment degradation products, such as ionones, damascenones, and megastigmatrienone, which are derived from carotenoid pigments unique to dry leaves. All of these aroma constituents are accumulated or dry-fixed inside cells. Further, exemplary secretion aroma constituents include diterpenes and sesquiterpenes, such as cembratrienediol (CBT) and abienol. CBT and so forth are substances that are present outside cells but retained near the surfaces by the constituents present inside the cells. Among these, particularly important constituents for specifying the surface migration rate X include the following.

[Table A]

	Constituent	Retention time when using nonpolar column (min)
Alkaloid	nicotine	24.95
Terpene degradation products (terpene aroma constituents)	solanone	25.66
	norsolanadione	29.47
Pigment degradation products	megastigmatrienone	32.17 to 33.49
	3-oxo-α-ionol	34.07
	neophytadiene	39.17
Accumulation in vacuole	scopoletin	42.04
Cuticle constituents (secretion aroma constituents)	cembratrienediol (CBT)	48.49
	heptacosane	57.42
	hentriacontane	64.19
Cell constituent	stigmasterol	67.43

[0041] At least one surface migration rate X for these constituents preferably exceeds 1. In one embodiment, the impregnation time is 10 minutes or more or 15 minutes or more.

[0042] After impregnation, the processed tobacco raw material is separated from a medium to end the impregnation step. The separation method is not limited, but a medium may be removed through evaporation. For example, when liquid or supercritical carbon dioxide is used, carbon dioxide can be removed by opening the reaction system to allow the pressure to return to atmospheric pressure. A tobacco material can thus be isolated. The time for releasing the pressure is not limited and may be set to 0.1 to 100 minutes, for example.

[0043] In the case of conducting the impregnation step using water or an organic solvent at atmospheric pressure, a tobacco raw material can be separated, for example, by evaporating water or the organic solvent. Further, the processed tobacco raw material need not necessarily be separated from a medium after impregnation. In this case, a tobacco material can be obtained as a mixture of the processed tobacco raw material and a medium.

(4) Pulverization Step

[0044] A separated tobacco material may be pulverized into a desirable size by a known method. Moreover, a tobacco material obtained as a mixture may also be subjected to pulverization. When liquid or supercritical carbon dioxide is used as a medium, the tobacco material is a mixture of the processed tobacco raw material and dry ice. Consequently, the tobacco raw material in the frozen state as it is can be subjected to freeze grinding. Alternatively, when a medium that is liquid at atmospheric pressure (water or organic solvent) is used, a tobacco material is obtained as a mixture of the processed tobacco raw material and the liquid. In this case, the tobacco material may be subjected to wet milling. In one embodiment, a pulverized tobacco material preferably has a D90 of 30 μm or less.

2. Tobacco Material

[0045] In a tobacco material obtained by a production method of the present invention (hereinafter, referred to as "tobacco material of the present invention"), an aroma constituent that has originally been present inside cells is present outside the cells or an aroma constituent that has originally been present outside cells is trapped by a constituent that has migrated from the inside of the cells, thereby being present on the surface in an increased amount. In other words, a tobacco material of the present invention is characterized by the surface migration rate X of more than 1. For this reason, when the tobacco material is used for a flavor inhaler article, an aroma constituent is readily released to provide favorable smoking flavor to a user. Such a flavor inhaler article will be described hereinafter.

[0046] A tobacco material of the present invention can be used in various forms, such as tobacco sheets, shreds, cigarette paper, or polysaccharide sheets, as described hereinafter.

(1) Tobacco Sheets

[0047] A tobacco sheet is a sheet obtained through forming of a composition containing a tobacco raw material. A tobacco sheet can be prepared from a composition containing tobacco leaves or laminae, which are a tobacco material of the present invention. In the present invention, a "sheet" indicates a material having pairs of almost parallel main surfaces and side surfaces. A tobacco sheet can be formed by a known process, such as a papermaking process, a casting process, or a rolling process. The details of various tobacco sheets formed by these processes are disclosed in "Tobacco no Jiten (Encyclopedia of Tobacco), Tobacco Academic Studies Center, March 31, 2009."

[0048] For example, in a papermaking process, a sheet can be produced by the steps of extracting water-soluble components from the tobacco leaves or laminae, separating into a water extract and residue, making a mixture of defibrated residue and pulp into a sheet, and adding a concentrate of the water extract to the sheet. In a casting process, a cast sheet can be produced by the steps of mixing water, pulp, a binder, and the tobacco leaves or laminae and casting the resulting mixture. In a rolling process, a sheet can be produced by the steps of mixing water, pulp, a binder, and the pulverized tobacco leaves or laminae and rolling the resulting mixture using a plurality of rolling rollers.

[0049] Further, as described in WO 2014/104078, a nonwoven fabric-like tobacco sheet can be obtained by mixing the pulverized tobacco leaves or laminae with a binder, sandwiching the resulting mixture by nonwoven fabrics, and forming the resulting laminate into a certain shape through heat fusing.

[0050] A tobacco sheet may contain an aerosol former. An aerosol former is not particularly limited and may be selected depending on the uses from various natural extract substances or the constituents thereof. Concrete examples of the aerosol former include: polyhydric alcohols, such as glycerol, propylene glycol, sorbitol, xylitol, and erythritol; triacetin; 1,3-butanediol; and mixtures thereof. The content of aerosol former may be adjusted to various amounts depending on the forms used in tobacco products. For example, when an aerosol former is contained in a tobacco sheet, the content is typically 5 weight% or more, preferably 10 weight% or more, more preferably 15 weight% or more and typically 50 weight% or less, preferably 40 weight% or less, and more preferably 25 weight% or less relative to the total weight of the tobacco sheet from a viewpoint of attaining a satisfactory flavor.

(2) Shreds

[0051] The tobacco leaves or laminae may be cut into tobacco shreds, or stems may be cut into stem shreds. The size is not limited, and examples include those having a width of 0.5 to 2.0 mm and a length of 3 to 10 mm. Shreds of this size are preferable in an embodiment of packing shreds described hereinafter. Other examples include strand-type shreds prepared by shredding processed tobacco leaves into a width of 0.5 to 2.0 mm and a length longer than the above-mentioned tobacco shreds and preferably comparable to the length of a cigarette paper.

[0052] Shreds may contain the above-mentioned aerosol former. When shreds contain an aerosol former, the content is typically 5 weight% or more, preferably 10 weight% or more, more preferably 15 weight% or more and typically 50 weight% or less, preferably 40 weight% or less, and more preferably 25 weight% or less relative to the weight of shreds from a viewpoint of generating a sufficient amount of aerosol and attaining a satisfactory flavor.

4. Tobacco Flavor Inhaler Articles

[0053] In the present invention, a "flavor inhaler article" indicates an article for inhaling a flavor by a user. Among such flavor inhaler articles, those including tobacco or a tobacco-derived component are referred to as "tobacco flavor inhaler articles." Tobacco flavor inhaler articles are broadly divided into "combustion-type tobacco flavor inhaler articles" (also simply referred to as "smoking articles") that generate a flavor through combustion and "non-combustion tobacco flavor inhaler articles" that generate a flavor without combustion. Further, non-combustion tobacco flavor inhaler articles are broadly divided into "heat-not-burn tobacco flavor inhaler articles" that generate a flavor through heating and "non-combustion non-heating tobacco flavor inhaler articles" that generate a flavor without heating. A tobacco flavoring agent of the present invention is suitable for combustion-type tobacco flavor inhaler articles or heat-not-burn tobacco flavor inhaler articles.

(1) Combustion-type Tobacco Flavor Inhaler Articles

[0054] A combustion-type tobacco flavor inhaler article may have a known structure. For example, a combustion-type tobacco flavor inhaler article may include a tobacco rod section and a filter. The tobacco rod section is preferably filled with a tobacco material of the present invention in the form of shreds.

(2) Heat-not-burn Tobacco Flavor Inhaler Articles

[0055] Fig. 1 illustrates an embodiment of a heat-not-burn tobacco flavor inhaler article. As illustrated, a heat-not-burn tobacco flavor inhaler article 20 includes a tobacco rod section 20A, a tubular cooling section 20B having a perforation on the perimeter, and a filter section 20C. The heat-not-burn tobacco flavor inhaler article 20 may include other members. The length in the axial direction of the heat-not-burn tobacco flavor inhaler article 20 is not limited but is preferably 40 to 90 mm, more preferably 50 to 75 mm, and further preferably 50 to 60 mm. Moreover, the perimeter length of the heat-not-burn tobacco flavor inhaler article 20 is preferably 16 to 25 mm, more preferably 20 to 24 mm, and further preferably 21 to 23 mm. In an exemplary embodiment, the length of the tobacco rod section 20A is 20 mm, the length of the cooling section 20B is 20 mm, and the length of the filter section 20C is 7 mm. The lengths of these individual members may be changed appropriately depending on production feasibility, required quality, and so forth. Fig. 1 illustrates an embodiment in which a first segment 25 is disposed, but only a second segment 26 may be disposed, without disposing the first segment 25, on the downstream side of the cooling section 20B.

1) Tobacco Rod Section 20A

[0056] In the tobacco rod section 20A, a tobacco material of the present invention in the form of shreds or tobacco sheets may be used as a tobacco filler 21. The method of packing the tobacco filler 21 within a cigarette paper 22 is not particularly limited. For example, the tobacco filler 21 may be wrapped in the cigarette paper 22, or the tobacco filler 21 may be packed within a tubular cigarette paper 22. When the shape of tobacco has a longitudinal direction as in a rectangle, tobacco may be packed with the longitudinal direction randomly aligned within the cigarette paper 22 or may be packed with the longitudinal direction aligned with the axial direction or a direction perpendicular to the axial direction of the tobacco rod section 20A. Moreover, a cigarette paper containing the above-mentioned tobacco flavoring agent of the present invention may be used as the cigarette paper 22. By heating the tobacco rod section 20A, tobacco components, an aerosol former, and water contained in the tobacco filler 21 are vaporized and inhaled.

2) Cooling Section 20B

[0057] The cooling section 20B is preferably formed of a tubular member. Such a tubular member may be a paper tube 23 of cylindrically processed cardboard, for example. Moreover, the cooling section 20B may be formed from a thin material sheet creased and then pleated, gathered, or folded to form channels. Such a material may be, for example, a sheet material selected from the group consisting of polyethylene, polypropylene, polyvinyl chloride, polyethylene terephthalate, polylactic acid, cellulose acetate, and aluminum foil. The total surface area of the cooling section 20B is appropriately adjusted in view of cooling efficiency and may be set to 300 to 1000 mm^2/mm. The cooling section 20B preferably has a perforation 24. By the presence of the perforation 24, external air is introduced into the cooling section 20B during inhalation. Consequently, a vaporized aerosol component generated through heating of the tobacco rod section 20A comes into contact with external air and liquefies due to the lowering temperature, thereby forming an aerosol. The diameter (cross-sectional length) of the perforation 24 is not particularly limited but may be 0.5 to 1.5 mm, for example. 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 formed on the perimeter of the cooling section 20B.

[0058] The cooling section 20B may have, for example, a rod shape of 7 to 28 mm in length in the axial direction. For example, the length in the axial direction of the cooling section 20B may be set to 18 mm. The cooling section 20B has a substantially circular cross-sectional shape in the axial direction, and the diameter may be set to 5 to 10 mm. For example, the diameter of the cooling section may be set to about 7 mm.

3) Filter Section 20C

[0059] The structure of the filter section 20C is not particularly limited, and the filter section 20C may comprise one or more filling layers. One or more cigarette papers may be wrapped around such a filling layer. The draw resistance of the filter section 20C can be changed appropriately by the amount, material, or the like of filler to be packed in the filter section 20C. For example, when the filler is cellulose acetate fibers, the draw resistance can be increased by increasing the amount of cellulose acetate fibers packed in the filter section 20C. When the filler is cellulose acetate fibers, the filling density of cellulose acetate fibers may be 0.13 to 0.18 g/cm^3. Herein, the draw resistance is a value measured by a draw resistance measuring device (trade name: SODIMAX from Sodim SAS).

[0060] The perimeter length of the filter section 20C is not particularly limited but is preferably 16 to 25 mm, more preferably 20 to 24 mm, and further preferably 21 to 23 mm. The length of the filter section 20C in the axial direction (horizontal direction in Fig. 1) can be selected from 4 to 10 mm to satisfy the draw resistance of 15 to 60 mmH_2O/seg. The length in the axial direction of the filter section 20C is preferably 5 to 9 mm and more preferably 6 to 8 mm. The cross-sectional shape of the filter section 20C is not particularly limited and may be circular, elliptic, or polygonal, for example. Further, a breakable capsule containing a flavor, a flavor bead, or a flavor may be directly added to the filter section 20C.

[0061] The filter section 20C may include a center hole section as the first segment 25. The center hole section comprises a first filling layer 25a having one or more hollow portions and an inner plug wrapper (inner cigarette paper) 25b that covers the filling layer. The center hole section acts to increase the strength of a mouthpiece section. The center hole section may lack the inner plug wrapper 25b and retain the shape through thermoforming. The filter section 20C may include a second segment 26. The second segment 26 comprises a second filling layer 26a and an inner plug wrapper (inner cigarette paper) 26b that covers the filling layer. The second filling layer 26a may be, for example, a rod of ø50 to ø10 mm in inner diameter formed by hardening highly densely packed cellulose acetate fibers added with 6 to 20 weight%, based on the weight of cellulose acetate, of a plasticizer including triacetin. Since the second filling layer has a high filling density of fibers, air and an aerosol flow only through the hollow portion and hardly flow within the second filling layer during inhalation. Moreover, the second filling layer inside the center hole segment is a fiber-filled layer. Consequently, a user rarely feels odd by touch from the outside during use.

[0062] The first filling layer 25a and the second filling layer 26a are joined with an outer plug wrapper (outer cigarette paper) 27. The outer plug wrapper 27 may be a cylindrical paper, for example. Moreover, the tobacco rod section 20A, the cooling section 20B, and the connected first filling layer 25a and second filling layer 26a are joined with a mouthpiece lining paper 28. These three members may be joined, for example, by applying a glue, such as a vinyl acetate-based glue, to the inner surface of the mouthpiece lining paper 28 and wrapping the lining paper around these members. These members may also be joined separately using a plurality of lining papers.

[0063] The combination of a heat-not-burn tobacco flavor inhaler article and a heating device for generating an aerosol is also particularly referred to as heat-not-burn tobacco flavor inhaler system. Fig. 2 illustrates an example of such a system. In the figure, a heat-not-burn tobacco flavor inhaler system includes a heat-not-burn tobacco flavor inhaler article 20 and a heating device 10 for heating the tobacco rod section 20A from the outside.

[0064] 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 tubular recess 16, and the heater 12 and the metal tube 13 are arranged at positions corresponding to the tobacco rod section 20A to be inserted thereinto. The heater 13 may be an electric resistance heater, and heating by the heater 12 is performed by supplying power from the battery unit 14 in accordance with instructions from the control unit 15, which controls temperature. Heat generated by the heater 12 is transferred to the tobacco rod section 20A through the metal tube 13 having a high thermal conductivity. Although the figure illustrates an embodiment in which the heating device 10 heats the tobacco rod section 20A from the outside, the heating device may heat from the inside. The heating temperature by the heating device 10 is not particularly limited but is preferably 400°C or lower, more preferably 150°C to 400°C, and further preferably 200° to 350°C. Herein, the heating temperature means the temperature of the heater in the heating device 10.

EXAMPLES

[0065] Hereinafter, the present invention will be described by means of working examples. In the present invention, a pressure inside a vessel is a gauge pressure unless otherwise noted.

[Example 1] Investigation of Impregnation Conditions and COz Removal Conditions

[0066] About 600 g as dry weight of flue-cured tobacco laminae produced in Brazil having the initial water content of 10.5 weight% were weighed, placed in a stainless steel mesh container, and set in a pressure vessel (volume of 9 L). The pressure vessel was then purged with carbon dioxide gas for 10 seconds. Subsequently, carbon dioxide gas was introduced into the pressure vessel to raise the pressure inside the pressure vessel to the impregnation pressure of 30 kg/cm^2 (2.9 MPa as gauge pressure). After terminating the supply of carbon dioxide gas, the supply of liquid carbon dioxide was started from the upper part of the pressure vessel. Liquid carbon dioxide was gradually supplied until all the thermocouples set in the upper portion, the middle portion, and the lowest portion of the tobacco layer indicated the saturation temperature of carbon dioxide gas at the impregnation pressure. A little liquid carbon dioxide bled from the bottom of the pressure vessel almost at the same time as the thermocouple in the lowest portion indicated the saturation temperature. At this point, the supply of liquid carbon dioxide was terminated. One minute after terminating the supply, the pressure inside the pressure vessel was released to atmospheric pressure over about 1 minute (Experiment Level 1) or about 90 minutes (Experiment Level 2) to completely remove carbon dioxide from the vessel through volatilization. Further, the processed tobacco was taken out from the pressure vessel.

[Example 2] Outward Migration Rate of Constituents

[0067] Five grams of the unprocessed laminae and the processed laminae in Example 1 were prepared respectively. With reference to the extraction method by Severson et al., each lamina was picked up with tweezers and immersed in 100 mL of ethyl acetate in a 300 mL glass container for 30 seconds or less to dissolve, if possible, constituents present only on the surface. Ethyl acetate was then removed under reduced pressure using a rotary evaporator, and the weight of the resulting dry solid was measured. The dry solid was completely dissolved in chloroform to prepare a solution of 4 weight% concentration and analyzed by GC/MS under the conditions in Table 1. Table 2 shows the surface migration rates. Fig. 3 shows the gas chromatograms. In accordance with the method by Severson et al., it was determined that each constituent migrated from inside to outside the cells of tobacco leaves when the surface migration rate exceeded 1.

[Table 1]

Stetting items	Setting conditions
Oven	40°C (3 min)→4°C/min→280°C (20 min)
Run time	83 min
Amount fed	1 μL
Feeding mode	split (10:1)
Inlet temperature	270°C
Septum purge flow rate	5 mL/min
Gas saver	OFF
Transfer line temperature	280°C
Column	HP-5MS (30 m*0.25 mm*0.25 μm)
Column flow rate	1 mL/min (constant flow mode)
Solvent waiting time	4 min
Measurement mode	SCAN

(continued)

Stetting items	Setting conditions
Mass range	26 to 450
Threshold	50 (scan/sec 3.39)
MS ion source temperature	230°C
MS quadrupole temperature	150°C

[Table 2-1]

Compound	Retention time (min)	Surface migration rate X					Target ion m/z
		unprocessed	level 1	t-test	level 2	t-test	
benzyl alcohol	13.78	1.00	1.27		1.45		108
phenylethyl alcohol	16.76	1.00	1.23		1.41		122
phenylacetic acid	22.00	1.00	1.73	*	2.28	**	136
nicotine	24.95	1.00	1.46	*	1.51	*	162
solanone	25.66	1.00	1.45	*	1.82	**	194
myosmine	27.38	1.00	1.43		1.77		146
p-hydroxyphenylethanol	27.62	1.00	1.40		1.98	**	138
geranylacetone	28.38	1.00	1.17		1.47	*	151
nicotine- 1'-oxide	29.03	1.00	1.17		0.90		119
norsolanadione	29.47	1.00	1.05		1.23		126
2,3'-dipyridyl	30.65	1.00	1.31		1.49		156
dihydroactinidiolide	30.71	1.00	1.18		1.29		111
megastigmatrienone	31.67	1.00	1.25		1.51	*	190
megastigmatrienone	32.17	1.00	1.15		1.25		148
megastigmatrienone	33.16	1.00	1.28		1.55	*	190
3-hydroxy- β-damascone	33.24	1.00	1.35		1.58	*	193
megastigmatrienone	33.49	1.00	1.18		1.33		148
3-oxo-α-ionol	34.07	1.00	1.17		1.39		152
3-hydroxy-5,6-epoxy-β-ionol	34.70	1.00	1.43		1.83	**	125
3-oxo-7,8-dihydro-3-oxo-α-ionol	35.64	1.00	1.28		1.60	*	108
cotinine	35.68	1.00	1.19		1.49		176
dehydrovomifoliol	37.94	1.00	1.26		1.56	*	124
neophytadiene	39.17	1.00	0.94		1.02		278
hexahydrofarnesyl acetone	39.32	1.00	1.07		1.18		58
p-value; **: p <0.01, *: p <0.05							

[Table 2-2]

Compound	Retention time (min)	Surface migration rate X					Target ion m/z
		unprocessed	level 1	t-test	level 2	t-test	
3-hydroxysolavetivone	40.28	1.00	1.13		1.29		176
scopoletin	42.04	1.00	1.40		1.80	**	164
palmitic acid	42.21	1.00	1.27		1.81	**	213
CBT	48.49	1.00	1.05		1.26		245
heptacosane	57.42	1.00	1.73		2.06	**	380
octacosane	59.16	1.00	2.06	**	2.54	**	85
2-methyloctacosane	60.24	1.00	1.68	*	2.12	**	365
nonacosane	60.87	1.00	2.13	**	2.77	**	408
2-methylnonacosane	61.90	1.00	2.60	**	3.43	**	379
3-methylnonacosane	62.08	1.00	1.59	*	1.88	**	393
triacontane	62.50	1.00	2.14	**	2.71	**	71
2-methyltriacontane	63.54	1.00	1.86	**	2.19	**	393
3-methyltriacontane	63.72	1.00	1.80	**	2.29	**	407
hentriacontane	64.19	1.00	1.90	**	2.47	**	99
α-tocopherol	64.86	1.00	1.22		1.47	*	430
2-methylhentriacontane	65.36	1.00	2.78	**	3.55	**	407
3-methylhentriacontane	65.61	1.00	1.70	*	2.05	**	421
dotriacontane	66.10	1.00	2.37	**	3.49	**	99
campesterol	66.70	1.00	1.02		1.33		289
stigmasterol	67.43	1.00	1.18		1.45	*	412
2-methyldotriacontane	67.53	1.00	2.04	**	2.65	**	99
3-methyldotriacontane	67.81	1.00	2.21	**	2.95	**	435
tritriacontane	68.45	1.00	2.37	**	3.55	**	99
p-value; **: p <0.01, *: p <0.05							

[Example 3] Investigation of Impregnation Conditions and CO_2 Removal Conditions

[0068] Representative laminae were placed in a stainless steel mesh container and set in a pressure vessel (volume of 1 L, diameter of 80 mm, depth of 200 mm). The pressure vessel was then purged with carbon dioxide gas for 10 seconds. Subsequently, carbon dioxide gas was introduced into the pressure vessel to raise the pressure inside the pressure vessel to the impregnation pressure of 30 or 50 kg/cm^2 (2.9 MPa and 4.9 MPa, respectively, as gauge pressure). After terminating the supply of carbon dioxide gas, the supply of liquid carbon dioxide was started from the upper part of the pressure vessel. Liquid carbon dioxide was gradually supplied until all the thermocouples set in the upper portion, the middle portion, and the lowest portion of the tobacco layer indicated the saturation temperature of carbon dioxide gas at the impregnation pressure. A little liquid carbon dioxide bled from the bottom of the pressure vessel almost at the same time as the thermocouple in the lowest portion indicated the saturation temperature. At this point, the supply of liquid carbon dioxide was terminated. The time for impregnating with liquid carbon dioxide after terminating the supply and the time for releasing the pressure inside the pressure vessel to atmospheric pressure are as shown in Table 3. After the vessel was opened, laminae impregnated with carbon dioxide were taken out therefrom. As in the foregoing, a surface migration rate X was obtained for each sample.

[Table 3]

						Surface migration rate X					
Level	Impregnation pressure (bar)	Initial water content (%)	Impregnation time (min)	Release time (min)	Amount of sample (g)	nicotine	solanone	norsolanadione	megastigmatrienone 1*	megastigmatrienone 2*	3-oxo-α-ionol
Comparative	0	10	0	0	5.03	1	1	1	1	1	1
5	30	10	1	1	5.01	0.36	0.68	1.44	1.25	0.78	1.21
6	30	10	1	15	5.09	0.62	0.96	0.89	1.70	1.10	1.71
7	30	10	1	30	5.04	0.92	1.16	1.11	1.64	1.15	1.80
8	30	10	1	60	5.05	0.67	0.87	0.82	1.41	1.03	1.37
9	30	10	1	90	5.02	0.93	1.26	1.19	2.58	1.50	2.67
10	50	10	1	1	5.10	0.75	1.48	1.38	1.76	1.44	1.63
11	50	10	1	15	5.04	1.25	0.68	0.68	1.92	1.66	1.93
12	50	10	1	30	5.07	1.43	1.20	1.19	1.84	1.74	2.24
13	50	10	1	60	5.03	1.53	2.29	1.89	1.53	1.44	1.85
14	50	10	1	90	5.02	1.88	1.68	1.44	2.83	2.59	2.27

						Surface migration rate X					
Level	Impregnation pressure (bar)	Initial water content (%)	Impregnation time (min)	Release time (min)	Amount of sample (g)	neophytadiene	scopoletin	CBT	heptacosane	hentriacontane	stigmasterol
Comparative	0	10	0	0	5.03	1	1	1	1	1	1
5	30	10	1	1	5.01	0.45	0.77	2.34	1.00	1.34	2.14
6	30	10	1	15	5.09	0.73	1.55	3.77	1.68	1.79	3.07
7	30	10	1	30	5.04	0.63	1.33	4.43	1.42	1.29	2.31
8	30	10	1	60	5.05	0.59	0.97	3.25	1.08	1.20	2.00
9	30	10	1	90	5.02	0.90	2.00	3.00	1.91	2.60	3.19
10	50	10	1	1	5.10	1.21	1.15	7.58	2.52	2.41	3.43

(continued)

						Surface migration rate X						
Level	Impregnation pressure (bar)	Initial water content (%)	Impregnation time (min)	Release time (min)	Amount of sample (g)	neophytadiene	scopoletin	CBT	heptacosane	hentriacontane	stigmasterol	
11	50	10	1	15	5.04	1.09	2.24	2.55	1.40	2.26	3.38	
12	50	10	1	30	5.07	1.58	1.37	4.88	1.64	2.28	4.15	
13	50	10	1	60	5.03	0.67	1.41	4.40	1.91	2.12	1.95	
14	50	10	1	90	5.02	2.82	2.27	4.00	0.99	2.35	5.50	

*isomer

[Example 4] Freeze Grinding

[0069] Into a stainless steel pressure vessel, 60 g of laminae of flue-cured tobacco (cutters of flue-cured tobacco produced in Brazil) were placed. Subsequently, the vessel was filled with carbon dioxide to have an internal pressure of 50 kg/cm^2 (4.9 MPa as gauge pressure) and left for one minute. The internal temperature was 15.1°C. The valve of the pressure vessel was then open to allow the pressure to return to atmospheric pressure over 30 minutes. At this point, the laminae had the water content and the dry ice content of 10 and 16 weight%, respectively. The laminae in the frozen state were pulverized using a lab mill (Power Mill LM-05 from Dalton Corporation) into fine powder having an average particle size of 30 μm or less.

[Example 5]

[0070] Into a stainless steel pressure vessel, 700 g of laminae of flue-cured tobacco (cutters of flue-cured tobacco produced in Brazil) were placed. Subsequently, the vessel was filled with carbon dioxide to have an internal pressure of 30 kg/cm^2 (2.9 MPa as gauge pressure) and left for one minute. The internal temperature was -4.4°C. The valve of the pressure vessel was then open to allow the pressure to return to atmospheric pressure over 90 minutes. At his point, the laminae had the water content of 10 weight%. The laminae were cut using a shredder into shreds having a width of about 1 mm and a length of about 5 to 20 mm. About 700 mg of the shreds were packed randomly aligning within a cylindrical cigarette paper having a circumference of 24.4 mm and a length of 50 mm to obtain a smoking article. For the smoking article, fully trained seven experts made smoking evaluations by the method described hereinafter.

[Example 6]

[0071] The laminae were processed in the same manner as Example 5 except for opening the valve of the pressure vessel to allow the pressure to return to atmospheric pressure over one minute. The processed laminae had the water content of 10 weight%. By using the laminae, a smoking article was obtained and evaluated in the same manner as Example 5.

[Comparative Example]

[0072] Laminae of flue-cured tobacco (cuttings of flue-cured tobacco produced in Brazil) were conditioned to have the water content of about 12 weight%. By using the laminae, a smoking article was obtained and evaluated in the same manner as Example 5. The results are shown in the table below.

[Table 4]

	Example 5	Example 6
Flavor	A	B
Sweetness	A	B
Astringency	D	C

[Smoking Evaluation]

[0073]

Number of puffs: 7 to 10
Smoking order: smoking the smoking article obtained in the Comparative Example, then, the smoking article obtained in Example 5, then, the smoking article obtained in the Comparative Example, and then, the smoking article obtained in Example 6
Evaluation items: flavor, sweetness, astringency

Score:		
	A	significant increase relative to the Comparative Example
	B	increase relative to the Comparative Example
	C	decrease relative to the Comparative Example
	D	significant decrease relative to the Comparative Example

REFERENCE SIGNS LIST

[0074]

10	Heating device
11	Body
12	Heater
13	Metal tube
14	Battery unit
15	Control unit
16	Recess
17	Ventilation hole

20	Heat-not-burn flavor inhaler article
20A	Tobacco rod section
20B	Cooling section
20C	Filter section

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

Claims

1. A method of producing a tobacco material, comprising a step of impregnating a tobacco raw material with a medium in the state selected from the group consisting of gas, liquid, and supercritical states to obtain a tobacco material having a surface migration rate X defined below of more than 1.

$$\text{Surface migration rate } X = S/P$$

S: a GC peak area for a flavor component present on the surface of a tobacco raw material after impregnation
P: a GC peak area for the flavor component present on the surface of the tobacco raw material before impregnation

2. The method according to Claim 1, wherein the medium is carbon dioxide, water, or an organic solvent.

3. The method according to Claim 1 or 2, wherein the medium is liquid carbon dioxide or supercritical carbon dioxide.

4. The method according to any of Claims 1 to 3, wherein the step of impregnating is conducted under a pressure of 2 MPa or more.

5. The method according to any of Claims 1 to 4, wherein the step of impregnating is conducted for 10 minutes or more.

6. The method according to any of Claims 1 to 5, wherein S and P are determined under the following conditions.
[Conditions]

1) Five grams of a tobacco raw material after impregnation is weighed.

2) Into a container, 100 mL of an aprotic solvent and then the weighed sample are fed.
3) Subsequently, the container is shaken for 30 seconds for extraction.
4) The resulting extract is filtered to obtain a filtrate.
5) The extract obtained in 4) is filtered.
6) The filtered extract is added with a desiccant and dehydrated overnight.
7) The desiccant is filtered out, and the resulting filtrate is concentrated under reduced pressure to obtain a dry solid. The dry solid is dissolved in the aprotic solvent to prepare a 4% solution and analyzed by GC/MS to obtain the S value.
8) The tobacco raw material before impregnation is subjected to the procedure of 1) to 7) to obtain the P value.

7. The method according to any of Claims 1 to 6, wherein the tobacco raw material is conditioned or unconditioned tobacco leaves, laminae, stems, stalks, or shreds.

8. The method according to any of Claims 1 to 7, further comprising a step of pulverizing the tobacco raw material after the impregnating.

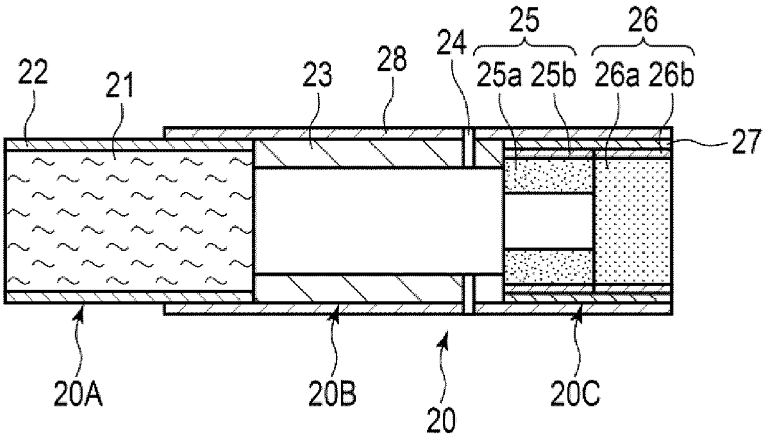
9. The method according to any of Claims 1 to 8, wherein the tobacco raw material is subjected to, after the impregnating, pulverization without separating from or after separating from the medium.

10. The method according to Claim 9, wherein the pulverization is freeze grinding.

11. A tobacco material obtained by the method of Claims 1 to 10.

12. The tobacco material according to Claim 11, having a D90 of 30 μm or less.

Fig. 1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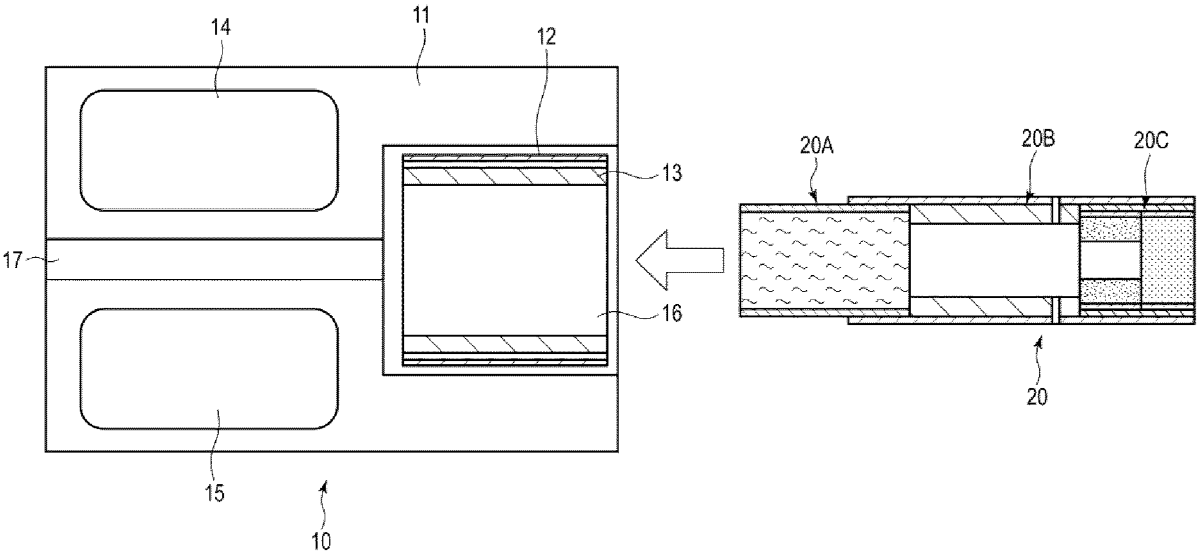
20B
20
20C

Fig. 2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20C
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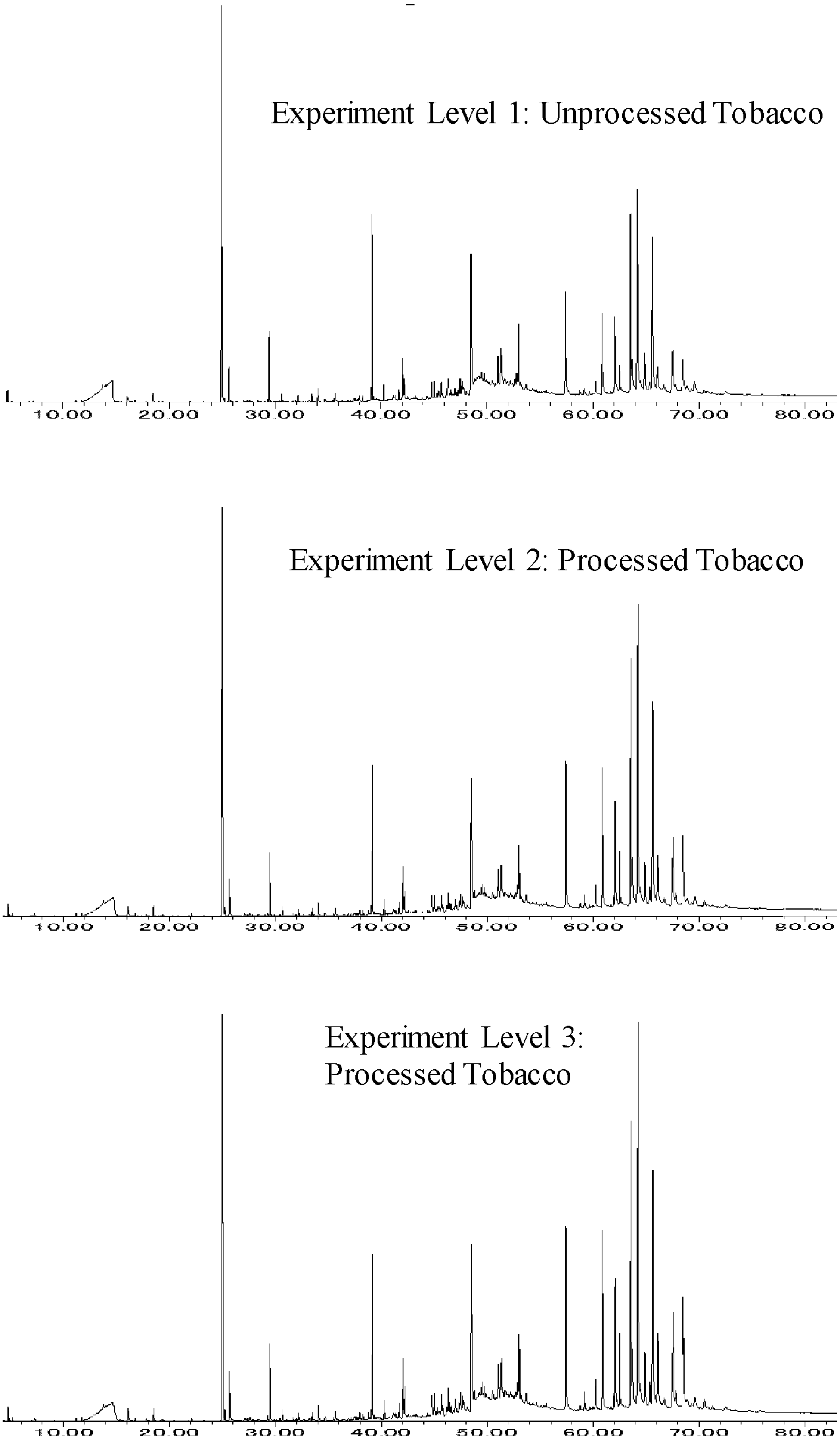
Fig. 3



Experiment Level 1: Unprocessed Tobacco

Experiment Level 2: Processed Tobacco

Experiment Level 3: Processed Tobacco

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2021/042191

A. CLASSIFICATION OF SUBJECT MATTER

A24B 15/16(2020.01)i; ***A24B 15/24***(2006.01)i
FI: A24B15/16; A24B15/24

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)

A24B15/16; A24B15/24

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.
X Y	JP 8-19389 A (JAPAN TOBACCO INC.) 23 January 1996 (1996-01-23) paragraphs [0008]-[0038], fig. 1, 2	1-6, 11 2, 7-10, 12
Y	JP 6-209756 A (JAPAN TOBACCO INC.) 02 August 1994 (1994-08-02) paragraph [0021]	2
Y	WO 2014/020699 A1 (JAPAN TOBACCO INC.) 06 February 2014 (2014-02-06) paragraphs [0014]-[0016], fig. 1	7-10
Y	WO 2019/049207 A1 (JAPAN TOBACCO INC.) 14 March 2019 (2019-03-14) paragraphs [0010]-[0012]	8-10, 12
Y	JP 63-167784 A (KOWA DISPLAY CO., INC.) 11 July 1988 (1988-07-11) page 2, lower left column, lines 7-14	10
A	WO 2017/141406 A1 (JAPAN TOBACCO INC.) 24 August 2017 (2017-08-24) paragraphs [0013]-[0015]	1-12

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

* Special categories of cited documents:
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Date of the actual completion of the international search	Date of mailing of the international search report
06 January 2022	**18 January 2022**

Name and mailing address of the ISA/JP	Authorized officer
Japan Patent Office (ISA/JP) **3-4-3 Kasumigaseki, Chiyoda-ku, Tokyo 100-8915** **Japan**	Telephone No.

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2021/042191

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2014-503199 A (SHANGHAI TOBACCO GROUP CO., LTD.) 13 February 2014 (2014-02-13) entire text, all drawings	1-12

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
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