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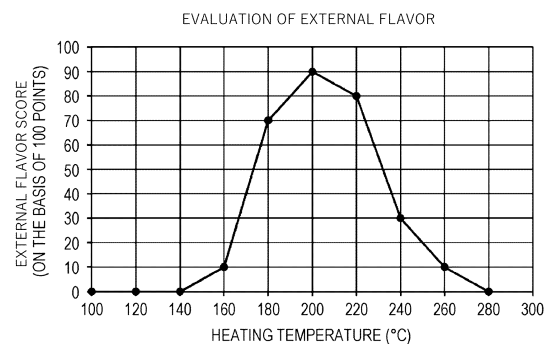
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(54) **PRODUCTION METHOD AND PRODUCTION APPARATUS FOR SOLUTION CONTAINING FLAVOR INGREDIENT FOR TOBACCO PRODUCT**

(57) Provided is a method for producing a solution containing a flavor ingredient for a tobacco product, the method comprising a step of bringing an aerosol composed of an aerosol-source material containing at least one of glycerin and propylene glycol into contact with a plant material heated to 180 to 220°C and a step of collecting the aerosol after contact with the plant material.

FIG. 2



Description

Technical Field

5 **[0001]** The present invention relates to a method for producing a solution containing a flavor ingredient for a tobacco product and to an apparatus for producing the solution.

Background Art

10 **[0002]** Various solutions containing a flavor ingredient are often used to change the flavor of tobacco products. Examples of the flavor ingredient include those derived from tobacco materials. Several methods have been proposed to obtain a solution containing a flavor ingredient derived from tobacco materials.

[0003] For example, Patent document 1 discloses a process for obtaining a tobacco-flavored extract. The process includes a step of performing conditioning by heating a tobacco mixture to a temperature of about 30°C to about 90°C for about 30 seconds to about 1 hour. Patent document 2 discloses a method for extracting volatile components from a tobacco material and a method for preparing a pre-vapor formulation containing the extracted volatile components. The methods include heating a tobacco material to a temperature range of about 50°C to about 250°C, recovering volatiles from the heated tobacco material, and combining the volatiles with a pre-vapor formulation after the recovering.

20 Citation List

Patent document

[0004]

25 Patent document 1: Japanese Unexamined Patent Application Publication (Translation of PCT Application) No. 2018-537082

Patent document 2: Japanese Unexamined Patent Application Publication (Translation of PCT Application) No. 2019-507592

30 Summary of Invention

Technical Problem

35 **[0005]** In the method of Patent document 1, the tobacco material is heated at a low temperature (about 30°C to about 90°C), and it is thus expected that the flavor ingredient contained in the tobacco material cannot be sufficiently recovered. In the methods of Patent document 2, the volatile components generated by heating the tobacco material are recovered by, for example, adsorbing or absorbing the volatile components on an absorbent such as an activated carbon absorbent or other microporous materials, or condensing the volatile components. In such methods, a separate absorbent may be required, or the volatile components may suffer a loss in the course of condensation. Furthermore, since the heating temperature is high, undesirable flavor ingredients may be generated.

40 **[0006]** Accordingly, it is an object of the present invention to provide a method for producing a solution containing a flavor ingredient for tobacco products in which a flavor ingredient derived from a plant material can be efficiently collected and in which an undesired flavor ingredient is not contained as much as possible, and to provide an apparatus for producing the solution. Solution to Problem

45 **[0007]** As a result of thorough studies, the present inventors have found that a solution containing a flavor ingredient for tobacco products in which a flavor ingredient derived from a plant material can be efficiently collected and in which an undesired flavor ingredient is not contained as much as possible can be provided by a method including a step of bringing an aerosol composed of an aerosol-source material containing at least one of glycerin and propylene glycol into contact with a plant material heated to 180°C to 220°C and a step of collecting the aerosol after contact with the plant material.

[0008] That is, the gist of the present invention is as follows.

55 [1] A method for producing a solution containing a flavor ingredient for a tobacco product includes a step of bringing an aerosol composed of an aerosol-source material containing at least one of glycerin and propylene glycol into contact with a plant material heated to 180°C to 220°C and a step of collecting the aerosol after contact with the plant material.

[2] In the method for producing a solution containing a flavor ingredient for a tobacco product according to [1], the

aerosol-source material is at least one selected from glycerin, propylene glycol, and a mixture thereof.

[3] In the method for producing a solution containing a flavor ingredient for a tobacco product according to [2], the aerosol-source material is a mixture of glycerin and propylene glycol.

[4] In the method for producing a solution containing a flavor ingredient for a tobacco product according to any one of [1] to [3], the plant material is at least one selected from a herbal material, a tobacco material, and a mixture thereof.

[5] In the method for producing a solution containing a flavor ingredient for a tobacco product according to [4], the plant material includes at least one selected from a mesophyll, a vein, a stem, a root, a flower, a seed, and a fruit of a plant.

[6] In the method for producing a solution containing a flavor ingredient for a tobacco product according to any one of [1] to [5], the plant material contains a flavor-developing aid.

[7] In the method for producing a solution containing a flavor ingredient for a tobacco product according to any one of [1] to [6], the plant material contains a polyol.

[8] In the method for producing a solution containing a flavor ingredient for a tobacco product according to any one of [1] to [7], the plant material is in a shredded state or a pulverized state.

[9] In the method for producing a solution containing a flavor ingredient for a tobacco product according to [8], the plant material is in a shredded state or is pulverized and then reconstituted into a granular or sheet shape.

[10] In the method for producing a solution containing a flavor ingredient for a tobacco product according to any one of [1] to [9], the aerosol brought into contact with the tobacco material has a temperature of 20°C or higher and lower than 180°C.

[11] An apparatus for producing a solution containing a flavor ingredient for a tobacco product includes:

a device for generating an aerosol from an aerosol-source material containing at least one of glycerin and propylene glycol;

a plant material-filled layer in which a plant material is disposed and through which the generated aerosol passes to bring the plant material and the aerosol into contact with each other;

a heater that heats the plant material-filled layer to 180°C to 220°C; and

a collecting device that collects the aerosol that has passed through the plant material-filled layer.

Advantageous Effects of Invention

[0009] According to the present invention, there can be provided a solution containing a flavor ingredient for tobacco products in which a flavor ingredient derived from a plant material can be efficiently collected and in which an undesired flavor ingredient is not contained as much as possible.

Brief Description of Drawings

[0010]

[Fig. 1] Fig. 1 illustrates the relationship between the heating temperature of a plant material (tobacco) and the amount of a flavor ingredient (nicotine) recovered.

[Fig. 2] Fig. 2 illustrates the relationship between the heating temperature of a plant material (tobacco) and the external flavor score.

[Fig. 3] Fig. 3 schematically illustrates one embodiment of a production apparatus according to the present invention.

Description of Embodiments

[0011] Hereafter, embodiments of the present invention will be described in detail. However, these descriptions are merely examples (representative examples) of the embodiments of the present invention. The present invention is not limited to these descriptions without departing from the spirit of the present invention.

[0012] In the present specification, the term "for tobacco products" refers to being used for so-called known cigarette products, heat-not-burn tobacco products, or electrically heated tobacco products.

[0013] In the present specification, when "to" is used to express numerical values or physical property values by sandwiching "to" between the values, the values before and after "to" are inclusive.

[0014] [Method for producing solution containing flavor ingredient for tobacco product]

[0015] A method for producing a solution containing a flavor ingredient for a tobacco product (hereafter also simply referred to as a production method of the present invention) according to an embodiment of the present invention includes a step of bringing an aerosol composed of an aerosol-source material containing at least one of glycerin and propylene glycol into contact with a plant material heated to 180°C to 220°C and a step of collecting the aerosol after contact with the plant material.

<Aerosol>

[0016] In the production method of the present invention, an aerosol composed of an aerosol-source material containing at least one of glycerin and propylene glycol is brought into contact with a plant material heated to a predetermined temperature. The aerosol can be generated by aerosolizing an aerosol-source material with an appropriate aerosol generating device.

[0017] The aerosol-source material is a liquid that generates an aerosol by being heated, and contains at least one of glycerin and propylene glycol. At least one of glycerin and propylene glycol refers to a material selected from glycerin alone, propylene glycol alone, and a mixture thereof. The total content of glycerin and propylene glycol in the aerosol-source material is preferably 90 wt% or more, more preferably 95 wt% or more, and particularly preferably 100 wt%. For the above percentages by weight, water as an impurity is not taken into account.

[0018] When the total content of glycerin and propylene glycol in the aerosol-source material is 100 wt%, the aerosol-source material is composed of glycerin alone, propylene glycol alone, or a mixture of glycerin and propylene glycol. In particular, the aerosol-source material is preferably a mixture of glycerin and propylene glycol. The mixing ratio of glycerin and propylene glycol in this case is not particularly limited. The mixing is preferably performed so that the weight of propylene glycol is 1 to 9 when the weight of glycerin is assumed to be 1. The mixing is more preferably performed so that the weight of propylene glycol is 1 to 2.5 when the weight of glycerin is assumed to be 1.

[0019] The aerosol generating device is not particularly limited as long as the aerosol-source material can be aerosolized. For example, the aerosol generating device includes a reservoir for storing a generating-source material, an electrical load for atomizing the aerosol-source material, a wick for drawing the aerosol-source material from the reservoir to the load, and an aerosol flow path through which the aerosol generated by atomizing the aerosol-source material flows and which is connected to a plant material-filled layer filled with a plant material described later.

[0020] The reservoir stores an aerosol-source material. The reservoir may accommodate a porous body such as a resin web or cotton, and the aerosol-source material may be impregnated in the porous body. In the reservoir, only the aerosol-source material may be stored without accommodating a porous body such as a resin web or cotton.

[0021] The load is a member for atomizing the aerosol-source material without combustion by electric power supplied from an appropriate power source. For example, the load may be constituted by a heating wire (coil) wound at a predetermined pitch. The load may be any element capable of atomizing the aerosol-source material to generate an aerosol. The load is, for example, a heating element or an ultrasonic generator. Examples of the heating element include a heating resistor, a ceramic heater, and an induction heater.

[0022] The temperature of the aerosol obtained by atomizing the aerosol-source material is normally 20°C or higher and lower than 290°C. In particular, the temperature is preferably 30°C or higher and more preferably 38°C or higher. The upper limit is preferably a temperature lower than the boiling point of the aerosol-source material. For example, assuming that glycerin is solely contained as the aerosol-source material, the upper limit may be lower than 290°C. Assuming that propylene glycol is solely contained as the aerosol-source material, the upper limit may be lower than 188°C. When the aerosol-source material is a mixture of glycerin and propylene glycol, the upper limit of the temperature of the atomized aerosol may be, for example, lower than 188°C, which is the boiling point of propylene glycol. The upper limit of the temperature of the aerosol may also be lower than 180°C.

[0023] The temperature of the aerosol is preferably set to be lower than the heating temperature of the plant material to prevent the temperature of the plant material from exceeding the set heating temperature.

[0024] The wick may be a liquid holding member that draws the aerosol-source material from the reservoir to the load using a capillary phenomenon, and may be made of, for example, glass fiber or porous ceramic.

[0025] The aerosol flow path is disposed downstream of the load, and is connected to a plant material-filled layer so as to be in contact with the plant material described later. The temperature of the aerosol can be determined by inserting a thermocouple, from the outside of the aerosol flow path connecting the aerosol generating device and the plant material-filled layer, at a position of the aerosol flow path immediately before the inlet of the plant material-filled layer. According to this method, the temperature of a mixture fluid of the aerosol and air is measured, but this temperature is defined as a temperature of the aerosol in the present specification.

[0026] In the production method of the present invention, an aerosol generated from the aerosol-source material is brought into contact with a plant material heated to a predetermined temperature. A flavor ingredient contained in the plant material can be transferred to the aerosol by bringing the aerosol into contact with the plant material having a predetermined temperature. The contact of the aerosol with the plant material can be performed by, for example, preparing a plant material-filled layer as described later and causing the aerosol to flow through the filled layer. For formation of the filled layer, a tubular container having an inlet and an outlet through which an aerosol flows can be filled with plant materials in various forms described later to obtain a filled layer. Examples of the sectional (cross-sectional) shape of the tubular container include a circle, an ellipse, and a polygon.

[0027] The contact of the aerosol with the plant material may be performed in an environment using a fluidized bed in which the aerosol is injected into the container from the bottom so that the plant material is frequently brought into

contact with the aerosol in the container.

[0028] The plant material to be brought into contact with the aerosol needs to be heated to 180°C to 220°C. If the temperature of the plant material is lower than 180°C, the flavor ingredient contained in the plant material is not sufficiently transferred to the aerosol. If the temperature of the plant material is higher than 220°C, undesired ingredients are transferred to the aerosol and the resulting solution containing a flavor ingredient has an undesired flavor.

[0029] When the aerosol is caused to flow through the plant material-filled layer, the temperature of a tobacco material can be adjusted by performing heating with a heater arranged around the filled layer.

[0030] In the case of a fluidized bed, the temperature of the tobacco material can be adjusted by controlling the temperature of the container in which the aerosol and the plant material come into contact with each other.

[0031] The production method of the present invention includes a step of collecting the aerosol after contact with the plant material. The step of collecting the aerosol may be, for example, a step of condensing the aerosol into a liquid and collecting the aerosol in a liquid state. To condense the aerosol into a liquid, the aerosol may be condensed by means of cooling the aerosol.

[0032] Examples of the cooling means include means using a cooling device with a publicly known refrigerant and means using a cooling agent such as liquid nitrogen or dry ice. The temperature may be about -200°C to 10°C.

[0033] In the step of collecting the aerosol, the system may be set to a negative pressure so that the aerosol that has been brought into contact with the plant material is efficiently collected in the collection step.

<Plant material>

[0034] In the production method of the present invention, the flavor ingredient contained in the plant material is transferred to the aerosol by bringing the aerosol into contact with the plant material. Examples of the plant material include tobacco materials, acai berry, alfalfa, allspice, annatto seed, apricot oil, basil, bee balm, wild bergamot, black pepper, blueberry, borage oil, northern bugweed, cacao, calamus root, catmint, catuaba, cayenne pepper, chaga, chervil, cinnamon, dark chocolate, potato skin, Chinese ginseng, ginkgo, St. John's wort, saw palmetto, green tea, black tea, black cohosh, cayenne, chamomile, amsonia, cocoa, cranberry, dandelion, grapefruit, honeybush, echinacea, garlic, evening primrose, feverfew, ginger, goldenseal, Japanese hawthorn, hibiscus flower, jiaogulan, kava, lavender, licorice, sweet marjoram, milk thistle, mint (menthe), oolong tea, orange, oregano, papaya, pennyroyal, peppermint, red clover, rooibos (red or green), rosehip, rosemary, sage, clary sage, savory, spearmint, spirulina, sorghum savory, spearmint, spirulina, sorghum bran high-tannin species, sorghum kernel high-tannin species, Japanese lacquer bran, goji berry, gotu kola, thyme, turmeric, bearberry, valerian, wild yam root, wintergreen, yacon root, yellow dock, yerba mate, yerba santa, bacopa monniera, ashwagandha, eggplant, sweet pepper, tomato, potato, capsicum, Chinese lantern plant, and marian thistle.

[0035] Among the above plant materials, allspice, allspice, black pepper, northern bugweed, calamus root, catmint, catuaba, cayenne pepper, chaga, chervil, cinnamon, Chinese ginseng, St. John's wort, green tea, black tea, black cohosh, cayenne, chamomile, amsonia, cocoa, honeybush, echinacea, feverfew, ginger, goldenseal, lavender, licorice, sweet marjoram, milk thistle, mint (menthe), oolong tea, oregano, pennyroyal, peppermint, red clover, rooibos (red or green), rosehip, rosemary, sage, clary sage, savory, spearmint, gotu kola, thyme, turmeric, valerian, wintergreen, yellow dock, yerba mate, yerba santa, bacopa monniera, ashwagandha, capsicum, Chinese lantern plant, and marian thistle correspond to herbal materials.

[0036] Among the above plant materials, the plant material is preferably at least one selected from the herbal materials, tobacco materials, and mixtures thereof. The plant material preferably includes at least one selected from the mesophyll, vein, stem, root, flower, seed, and fruit of the above plant.

[0037] In addition, grape seeds, beet roots, slippery elm barks, comfrey leaves and roots, wild yam roots, and yacon roots can also be used.

[0038] When a tobacco material is used as the plant material, various types of tobaccos can be used. Examples of the types of tobaccos include yellow varieties, burley varieties, oriental varieties, native varieties, other *Nicotiana tabacum* varieties, *Nicotiana rustica* varieties, and mixtures of the foregoing. For example, the lamina, midrib, stem, flower, or root of these types of tobaccos can also be used. When a tobacco material is used as the plant material, nicotine can be exemplified as a main flavor ingredient.

[0039] These plant materials are preferably in a shredded state or a pulverized state from the viewpoint of increasing the surface area in contact with the aerosol. The plant material in a shredded state is a plant material shredded by appropriate means. For the size, the width of the shredded plant material may be, for example, 0.5 mm or more and 2.0 mm or less. The length of the shredded plant material may be in the range of about 0.5 mm or more and about 30.0 mm or less.

[0040] The plant material in a pulverized state is a plant material pulverized by appropriate means so that individual pieces of the pulverized plant material have a size smaller than that of the above-described shredded plant material. The pulverized plant material preferably has an average particle diameter of, for example, 200 μm or more and 300 μm

or less. The average particle diameter can be measured using a particle size analyzer (e.g., Mastersizer manufactured by Spectris Co., Ltd.). The pulverization may be performed through a plurality of steps such as coarse pulverization followed by fine pulverization. When a plurality of plant materials are used in a mixed manner, the mixed state of the plant materials can be easily made uniform by a method in which the plant materials are coarsely pulverized, then mixed with each other, and finely pulverized. The average particle diameter after the coarse pulverization can be set to, for example, 300 μm or more and 2 mm or less. The average particle diameter after the coarse pulverization can be measured using a multi-stage screen. A plant material after coarse pulverization can also be used without performing a fine pulverization step.

[0041] The above-described plant material in a shredded state or in a pulverized state may be a material processed by reconstituting a material into a sheet shape described later. In this case, the size can be set in the same range as described above.

[0042] The plant material preferably further contains a polyol. When the plant material further contains a polyol, the flavor ingredient contained in the plant material can be more efficiently transferred to the aerosol. This is because the partial pressure of the polyol in the plant material-filled layer increases during extraction when the polyol is contained, which facilitates volatilization of the flavor ingredient to increase the extraction efficiency. Preferably, the polyol is externally added to the plant material. Examples of the polyol include glycerin, propylene glycol, and 1,3-butanediol. A combination of glycerin and propylene glycol is preferred. The polyol is preferably added in an amount of 1 to 20 wt% and more preferably 5 to 10 wt% relative to the dry weight of the plant material when any of the polyols exemplified above or a mixture thereof is used.

[0043] The plant material preferably containing a polyol is the same in any of a shredded state, a pulverized state, a granular form, or a sheet form described later.

[0044] A flavor-developing aid can be added to the plant material. The flavor-developing aid may contain at least one of a carbonate, a hydrogencarbonate, an oxide, and a hydroxide of an alkali metal and/or an alkaline earth metal. The flavor-developing aid is preferably potassium carbonate or sodium carbonate. By adding the flavor-developing aid, plant components including amines are sufficiently volatilized, which can increase the yield of the flavor ingredient.

[0045] The pH of the plant material may be 7 to 9 through addition of the flavor-developing aid.

[0046] The pH can be measured using a pH meter (e.g., IQ240 manufactured by IQ Scientific Instruments Inc.) as follows. For example, distilled water is added to 2 to 10 g of the plant material in an amount 10 times the weight of the plant material. The mixture of water and the plant material is shaken at 200 rpm at room temperature (e.g., 25°C) for 10 minutes and allowed to stand for 5 minutes. Then, the pH of the extracted liquid is measured with a pH meter.

[0047] Furthermore, sugars, salts, and amino acids can also be contained as the flavor-developing aid. When such a component is contained as the flavor-developing aid, not only the volatilization of the component but also the reaction of the component readily occur upon heating the plant material to a treatment temperature according to an embodiment of the present application. Thus, a suitable flavor ingredient can be volatilized.

[0048] The amount of the flavor-developing aid added is preferably 1 to 20 wt% and more preferably 5 to 10 wt% relative to the dry weight of the plant material.

[0049] In at least one of exemplary embodiments, the tobacco material may be moisturized or treated with an additive before heating, or may be in the form of slurry or wet tobacco block. The additive may contain at least one of a solvent, a flavor-developing aid, and a flavor agent. A flavor-developing aid may be added to the tobacco material to release a flavor agent bonded to a salt, to hydrolyze a precursor of a flavor agent, or to perform both the release and the hydrolysis. The flavor-developing aid can also accelerate the reaction of components contained in the plant. The reaction is, for example, a Maillard reaction.

[0050] The plant material preferably containing a flavor-developing aid is the same in any of a shredded state, a pulverized state, or a sheet form described later. In the form of tobacco granules described later, an embodiment in which a flavor-developing aid is contained as a component (c) can be exemplified.

[0051] The plant material is preferably a plant material that is pulverized and then reconstituted into a granular or sheet shape.

<Sheet>

[0052] The sheet can be appropriately produced by a publicly known method such as a paper-making method, a slurry method, or a rolling method. All of the methods described below can be suitably applied when tobacco is used as the plant material.

[0053] In the case of the paper-making method, the sheet can be produced by a method including the following processes. 1) The plant material is coarsely pulverized and extracted with water to cause separation into a water extract and a residue. 2) The water extract is concentrated by vacuum drying. 3) Pulp is added to the residue, and the mixture is fiberized with a refiner and then subjected to paper-making. 4) A concentrated liquid of the water extract is added to the sheet obtained by paper-making, and drying is performed to form a sheet.

[0054] In the case of the slurry method, the sheet can be produced by a method including the following processes. 1) Water, a pulp, and a binder are mixed with a pulverized plant material. 2) The resulting mixture is thinly stretched (cast) and dried.

[0055] As described in International Publication No. 2014/104078, a nonwoven fabric sheet produced by a method including the following processes can also be used. 1) A powdery or granular plant material is mixed with a binder. 2) The resulting mixture is sandwiched between nonwoven fabrics. 3) The resulting laminate is formed into a predetermined shape by thermal welding to obtain a nonwoven fabric sheet. The plant material in this case is, for example, a tobacco.

[0056] The composition of the sheet is not particularly limited, but the content of the plant material is preferably, for example, 50 to 95 wt% relative to the total weight of the sheet. The sheet may contain a binder. Examples of the binder include guar gum, xanthan gum, CMC (carboxymethyl cellulose), and CMC-Na (sodium salt of carboxymethyl cellulose). The content of the binder is preferably 1 to 20 wt% relative to the total weight of the sheet. The sheet may further contain other additives. Examples of the additives include fillers such as pulp. The content of the filler is not particularly limited, but is preferably 1 wt% or more and 10 wt% or less relative to the total weight of the tobacco sheet.

[0057] The sheet may also contain a polyol. Examples of the polyol include glycerin, propylene glycol, and 1,3-butanediol, with a combination of glycerin and propylene glycol being preferred. The polyol is preferably added in an amount of 1 to 20 wt% relative to the dry weight of the sheet when any of the polyols exemplified above or a mixture thereof is used. The partial pressure of the polyol in the plant material-filled layer increases during extraction when the polyol is contained, which facilitates volatilization of the components in the sheet to increase the extraction efficiency.

<Granule>

[0058] The plant material may be in a granular state. Hereafter, this may also be simply referred to as granules.

[0059] The raw materials for the granules are, for example, (a) a pulverized plant material, (b) water, (c) at least one flavor-developing aid selected from the group consisting of potassium carbonate and sodium hydrogen carbonate, and (d) at least one binder selected from the group consisting of pullulan and hydroxypropyl cellulose. The plant material is preferably a tobacco.

[0060] The pulverized plant material (component (a)) contained as a raw material for the granules includes a plant material and the above sheet subjected to pulverization. When the plant material is a tobacco material, the types of tobaccos are burley varieties, yellow varieties, and oriental varieties. The pulverized plant material is preferably being pulverized to a size of 20 μm or more and 300 μm or less. The average particle diameter can be measured using a particle size analyzer (e.g., Mastersizer manufactured by Spectris Co., Ltd.).

[0061] The water (component (b)) contained in the granules is for maintaining the gathering of the granules.

[0062] The raw material mixture of the granules normally contains water in an amount of 3 wt% or more and 13 wt% or less. The granules may contain water in such an amount that the loss on drying is normally 5 wt% or more and 17 wt% or less. The loss on drying refers to a change in weight before and after drying when a sample is partly collected for measurement and the sample is completely dried by evaporating the whole water in the collected sample (e.g., when the sample is dried at a constant temperature (105°C) for 15 minutes). Specifically, the loss on drying refers to a ratio (wt%) of the sum of the amount of water contained in the sample and the amount of volatile components volatilized under the above drying conditions to the weight of the sample. That is, the loss on drying (wt%) can be represented by the following formula (1).

[0063] Loss on drying (wt%) = {(Weight of sample before complete drying) - (Weight of sample after complete drying)} $\times$ 100/Weight of sample before complete drying (1)

[0064] The flavor-developing aid (component (c)) that is optionally contained in the granules may be those exemplified above. Such a flavor-developing aid facilitates the release of the flavor ingredient contained in the granules from the granules, which provides a smoke taste that can satisfy users.

[0065] The raw material mixture of the granules may normally contain the flavor-developing aid in an amount of 5 wt% or more and 20 wt% or less.

[0066] The binder (component (d)) contained in the granules binds the raw materials for the granules to maintain the gathering of the granules. The binder is composed of pullulan, gellan gum, carrageenan, agar, guar gum, locust bean gum, hydroxypropyl cellulose (HPC), hydroxypropyl methylcellulose (HPMC), carboxymethyl cellulose (CMC), starch, modified starch, or a mixture thereof.

[0067] The raw material mixture of the granules may normally contain the binder in an amount of 0.5 wt% or more and 15 wt% or less.

[0068] The granules can be composed of the components (a), (b), (c), and (d) described above, but may further contain additional components.

[0069] When the granules are composed of the above components (a), (b), (c), and (d), the raw material mixture of the granules may normally contain the component (a) in an amount of about 20 wt% or more (about 80 wt% or less).

[0070] The granules can be obtained, for example, by mixing the components (a), (c), and (d), adding the component

(b) to the mixture, kneading the mixture, granulating the kneaded product (into a long column shape) with a wet extrusion granulator, and then sizing the granules into a short column shape or a spherical shape.

[0071] For the extrusion granulation, the kneaded product is preferably extruded at ambient temperature at a pressure of 2 kN or more. Through this high-pressure extrusion, the temperature of the kneaded product is rapidly increased in a moment at the outlet of the extrusion granulator from ambient temperature to, for example, 90°C or higher and 100°C or lower. As a result, water and volatile components are evaporated in an amount of 2 wt% or more and 4 wt% or less. Therefore, water to be added for preparing the kneaded product can be used in an amount larger than the desired water content in tobacco granules to be obtained by the above amount of water evaporated.

[0072] The granules obtained by the extrusion granulation may be further optionally dried for adjusting the water content. For example, the loss on drying of the granules obtained by extrusion granulation is measured. If the measured loss on drying is higher than the desired loss on drying (e.g., 5 wt% or more and 17 wt% or less), the tobacco granules may be further dried to achieve the desired loss on drying. The drying conditions (temperature and time) for achieving the desired loss on drying can be set based on drying conditions (temperature and time) determined in advance and required for reducing the loss on drying by a predetermined value.

[Apparatus for producing solution containing flavor ingredient]

[0073] An apparatus for producing a solution containing a flavor ingredient for tobacco products according to the present invention includes a device for generating an aerosol from an aerosol-source material containing at least one of glycerin and propylene glycol, a plant material-filled layer in which a plant material is disposed and through which the generated aerosol passes to bring the plant material and the aerosol into contact with each other, a heater that heats the plant material-filled layer to 180°C to 220°C, and a collecting device that collects the aerosol that has passed through the plant material-filled layer. Fig. 3 schematically illustrates an embodiment of the production apparatus. An aerosol generating device 1, a plant material-filled layer 4, and a collecting device 5 are connected so that an aerosol 3 generated by the aerosol generating device 1 passes through the plant material-filled layer 4 heated with a heater 2, and the aerosol 3 having passed through the plant material-filled layer 4 is collected by the collecting device 5.

[0074] An example of the device for generating an aerosol from an aerosol-source material containing at least one of glycerin and propylene glycol is the aerosol generating device having the configuration described in the above production method. The structural elements described in the above production method can be used for the corresponding structural elements of the aerosol generating device.

[0075] The plant material is disposed in the plant material-filled layer. Various materials described in the above production method can be used as a plant material to be subjected to filling. The plant material is preferably a tobacco.

[0076] The plant material is preferably brought into contact with the aerosol in a shredded state, in a pulverized state, in a granular form, or in a sheet form. The filled layer can be formed in any of the forms. The aerosol is preferably caused to flow through the filled layer.

[0077] The size and shape of the filled layer are not particularly limited. The size and shape of the filled layer may be dependent on the size and shape of a container to be filled with the plant material. An example of the container to be filled with the plant material is a tubular container provided with an inlet and an outlet through which an aerosol flows on a bottom surface and an upper surface of the tubular container, respectively. Examples of the sectional (cross-sectional) shape of the tubular container include a circle, an ellipse, and a polygon.

[0078] When the plant material constituting the plant material-filled layer is in a shredded state, in a pulverized state, or in the form of granules prepared from the plant material in the foregoing state, the filling density of the plant material is normally 150 mg/cm³ or more and preferably 250 mg/cm³ or more, and normally 600 mg/cm³ or less and preferably 350 mg/cm³ or less. Although depending on the shape of the plant material, an excessively high filling density prevents the aerosol from flowing through the filled layer. If the filling density is excessively low, the amount of an object to be extracted is inefficiently small with respect to the size of the container.

[0079] The plant material-filled layer is heated to 180°C to 220°C with a heater. The heater can directly or indirectly heat the plant material-filled layer. For example, the heater may be a sheet heater, an infrared heater, or an IH heater. The sheet heater is a flexible sheet-shaped heater and is, for example, a ribbon heater that generates heat by electric resistance. When the IH heater is used, heating can be performed not only from the metal container into which the filled layer is inserted but also from the inside of the filled layer by mixing a susceptor such as a metal powder in the plant material-filled layer.

[0080] When the outer peripheral surface of the plant material-filled layer is heated, the above-described sheet heater, infrared heater, or IH heater can be used. When heating is performed from the inside of the plant material-filled layer, the IH heater or the like can be used. In this case, the aerosol is preferably allowed to efficiently pass through the inside of the plant material-filled layer during extraction operation because the extraction efficiency is improved.

[0081] The setting temperature of the heater is not particularly limited as long as the plant material-filled layer is heated to 180°C to 220°C. The temperature of the plant material-filled layer (product temperature) can be measured by inserting

a thermometer into the plant material-filled layer.

[0082] When the aerosol generated with the aerosol generating device passes through the inside of the plant material-filled layer heated to 180°C to 220°C, the plant material and the aerosol come into contact with each other. Then, the flavor ingredient contained in the plant material is transferred to the aerosol. The aerosol is collected with the collecting device described later.

[0083] The aerosol collecting device collects the aerosol that has passed through the plant material-filled layer. The aerosol collecting device is, for example, a device including means for cooling an aerosol and a container for accommodating a liquid condensed by the cooling. Such a device is, for example, a so-called cold trap. In the cold trap, the pressure in the system is preferably reduced to a negative pressure by using, for example, an evaporator, but the negative pressure achieved by reduction in pressure is not necessarily required. The means for cooling an aerosol is not particularly limited. Examples of the means for cooling an aerosol include a method using a cooling device with a publicly known refrigerant and a method using a cooling agent such as liquid nitrogen or dry ice. The temperature during cooling may be about -200°C to 10°C. The aerosol condensed into a liquid is collected in a storing solution included in the collecting device.

[Examples]

<Experimental Example 1>

[0084] An aluminum pipe having an inside diameter of 7.2 mm was filled with 250 mg of a tobacco raw material composed of a shredded product of a sheet tobacco (a shredded product of a sheet obtained by paper-making, with a thickness of about 300 μm, a shredding width of 0.8 mm, and a length of about 0.5 to 30.0 mm; the raw materials of the sheet were 66 wt% of pulverized tobacco, 7 wt% of wood pulp, 15 wt% of glycerin, 12 wt% of water, and 1.32 wt% of nicotine) by a length of 20 mm to form a tobacco-filled layer. The aluminum pipe was inserted into an electric furnace having an inside diameter of 9.2 mm (manufactured by Ouji Shokai Co.) and heated from the outer periphery. The heating temperature was controlled with a temperature controller (Temperature controller TJA-50 manufactured by AS ONE Corporation). When the temperature reached a predetermined temperature, a predetermined carrier shown in Table 1 was allowed to flow through the tobacco-filled layer at a flow rate of 55 mL/2 sec. In Examples 1 to 3, the temperature of the aerosol allowed to flow through the tobacco-filled layer was 30°C. The temperature of the aerosol was determined by inserting a thermocouple, from the outside of a pipe connecting the aerosol generating device and the tobacco-filled layer, at a position of the pipe immediately before the inlet of the tobacco-filled layer and by measuring the temperature of a mixture fluid of the aerosol and air.

[0085] In Examples 1 to 3, the experiment was performed while the aerosol concentration in the carrier was adjusted to be substantially uniform. The air, vapor, or aerosol that had flowed through the tobacco-filled layer was trapped in a tube placed in an ice bath, and the liquid was recovered and analyzed.

[0086] Nicotine contained in the recovered liquid was quantified using a GC-MS (7890B manufactured by Agilent). Nicotine in the tobacco-filled layer was quantified by extracting a tobacco filling with a solvent (methanol) and then using a GC-MS (7890B manufactured by Agilent). Table 1 shows the recovery of nicotine for each carrier and each temperature. In conclusion, it was found that nicotine could be recovered most efficiently when an aerosol of propylene glycol and glycerin was caused to flow as compared with air.

[0087] The recovery of nicotine was calculated from the following formula using the weight of nicotine in the tobacco-filled layer before treatment and the weight of nicotine in the tobacco-filled layer after treatment.

Nicotine recovery (%)

$$= 100 - (\text{Weight of nicotine in tobacco-filled layer after treatment} / \text{Weight of nicotine in tobacco-filled layer before treatment}) \times$$

100

[Table 1]

Experimental Example	Heating temperature of plant material	Carrier passage time [min]	Carrier	Flavor ingredient (nicotine) recovery [%]
Comparative Example 1	200	10	Air	46.6
Comparative Example 2	200	4	Propylene glycol vapor	35.5
Example 1	200	4	Aerosol (propylene glycol and glycerin with a weight ratio of 1:1)	59.1
Example 2	200	4	Aerosol (propylene glycol)	53.7
Example 3	200	4	Aerosol (glycerin)	57.0

[0088] Fig. 1 shows the relationship between the heating temperature of the filled layer and the amount of nicotine recovered when each carrier was used. The horizontal axis represents the heating temperature of the filled layer, and the vertical axis represents the amount of nicotine recovered. The results in Fig. 1 show that the amount of nicotine recovered increases when the temperature of the filled layer is 180°C or higher.

[0089] The filled layer used in each test was prepared by filling the same tobacco raw material in the same filling amount. The amount of nicotine recovered is the weight of nicotine in a liquid trapped in the ice-cooled tube. Since the nicotine extracted from the tobacco-filled layer partially adheres to the inner wall of the pipe connecting the ice-cooled tube serving as the collecting device and the filled layer, all of the nicotine extracted from the tobacco-filled layer was not measured as the amount of nicotine recovered in this experiment.

<Experimental Example 2: Determination of external flavor score>

[0090] In the same manner as in Experimental Example 1, a condensed liquid was recovered by causing the same carrier as in Example 1 to pass through the filled layer heated to each temperature. One gram of the condensed liquid was inserted into a screw tube, and the persons handling the experiment (n = 3) gave an external flavor score. The score was given in increments of 10 points on the basis of 100 points, and the average of the scores was defined as an external flavor score. Fig. 2 illustrates the results. Those having a high score had a tobacco-like fragrance as an external flavor. Those having a low score were found when an unpleasant smell due to carbonization was produced.

[0091] Thus, it was confirmed that an unpleasant burnt smell was produced as the temperature increased to higher than 220°C. Therefore, it was found that an aerosol of propylene glycol and glycerin was desirably passed through a plant material-filled layer heated to 200 ± 20°C in order to efficiently achieve a large amount of flavor ingredient (nicotine) recovered and a good flavor ingredient.

Reference Signs List

[0092]

- 1 aerosol generating device
- 2 heater
- 3 aerosol
- 4 plant material-filled layer
- 5 collecting device

Claims

1. A method for producing a solution containing a flavor ingredient for a tobacco product, comprising a step of bringing an aerosol composed of an aerosol-source material containing at least one of glycerin and propylene glycol into contact with a plant material heated to 180°C to 220°C and a step of collecting the aerosol after contact with the plant material.

2. The method for producing a solution containing a flavor ingredient for a tobacco product according to claim 1, wherein the aerosol-source material is at least one selected from glycerin, propylene glycol, and a mixture thereof.
3. The method for producing a solution containing a flavor ingredient for a tobacco product according to claim 2, wherein the aerosol-source material is a mixture of glycerin and propylene glycol.
4. The method for producing a solution containing a flavor ingredient for a tobacco product according to any one of claims 1 to 3, wherein the plant material is at least one selected from a herbal material, a tobacco material, and a mixture thereof.
5. The method for producing a solution containing a flavor ingredient for a tobacco product according to claim 4, wherein the plant material includes at least one selected from a mesophyll, a vein, a stem, a root, a flower, a seed, and a fruit of a plant.
6. The method for producing a solution containing a flavor ingredient for a tobacco product according to any one of claims 1 to 5, wherein the plant material contains a flavor-developing aid.
7. The method for producing a solution containing a flavor ingredient for a tobacco product according to any one of claims 1 to 6, wherein the plant material contains a polyol.
8. The method for producing a solution containing a flavor ingredient for a tobacco product according to any one of claims 1 to 7, wherein the plant material is in a shredded state or a pulverized state.
9. The method for producing a solution containing a flavor ingredient for a tobacco product according to claim 8, wherein the plant material is in a shredded state or is pulverized and then reconstituted into a granular or sheet shape.
10. The method for producing a solution containing a flavor ingredient for a tobacco product according to any one of claims 1 to 9, wherein the aerosol brought into contact with the tobacco material has a temperature of 20°C or higher and lower than 180°C.
11. An apparatus for producing a solution containing a flavor ingredient for a tobacco product, comprising:
 - a device for generating an aerosol from an aerosol-source material containing at least one of glycerin and propylene glycol;
 - a plant material-filled layer in which a plant material is disposed and through which the generated aerosol passes to bring the plant material and the aerosol into contact with each other;
 - a heater that heats the plant material-filled layer to 180°C to 220°C; and
 - a collecting device that collects the aerosol that has passed through the plant material-filled layer.

FIG. 1

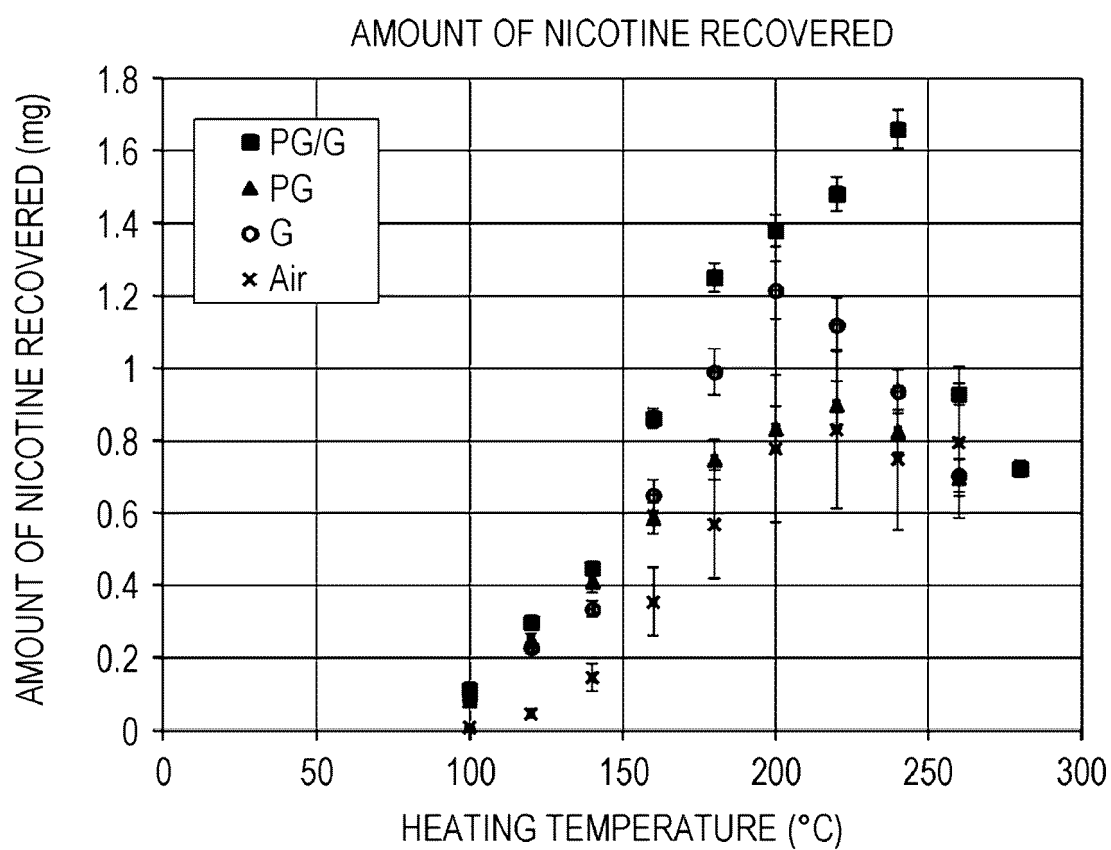


FIG. 2

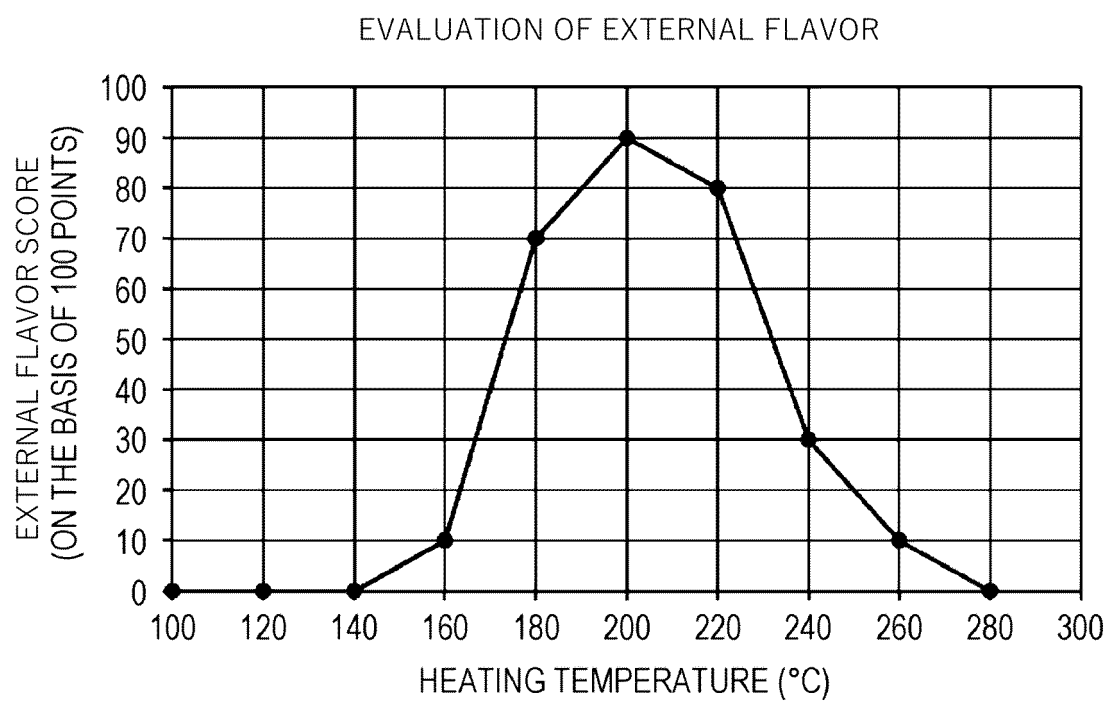
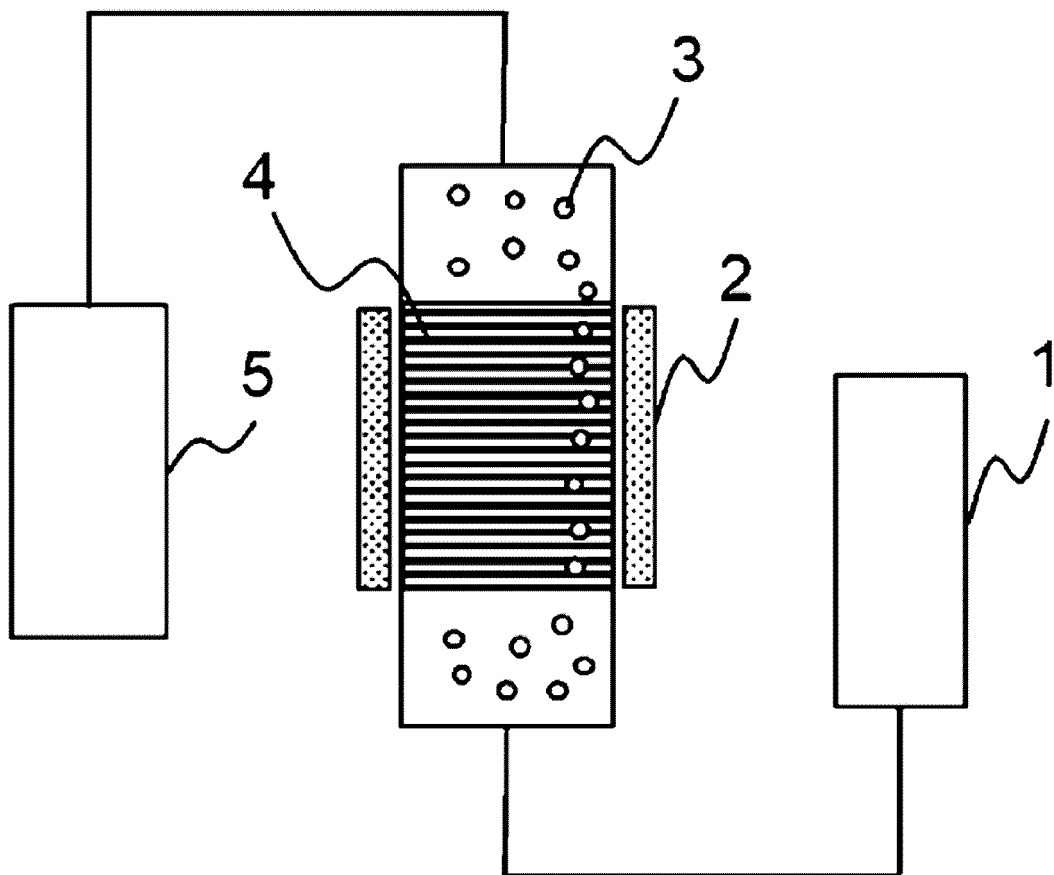


FIG. 3



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2021/028547

A. CLASSIFICATION OF SUBJECT MATTER

B01D 8/00(2006.01); **A24B 15/167**(2020.01); **A24B 15/24**(2006.01);
FI: A24B15/24; A24B15/167; B01D8/00 Z

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)

B01D8/00; A24B15/167; 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.
A	WO 2015/129098 A1 (JAPAN TOBACCO INC.) 03 September 2015 (2015-09-03) entire text, all drawings	1-11
A	JP 2020-519274 A (BRITISH AMERICAN TOBACCO (INVESTMENTS) LTD.) 02 July 2020 (2020-07-02) entire text, all drawings	1-11

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

* Special categories of cited documents:	"I" 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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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/JP2021/028547

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					entire text, all drawings		
				EP	3097793	A1	
				KR	10-2016-0110996	A	
JP	2020-519274	A	02 July 2020	US	2021/0153544	A1	
					entire text, all drawings		
				WO	2018/210677	A1	
				KR	10-2019-0134790	A	
				CN	110678090	A	

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REFERENCES CITED IN THE DESCRIPTION

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