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(54) **METHOD FOR MANUFACTURING TOBACCO RAW MATERIAL, AND TOBACCO RAW MATERIAL**

(57) Provided is a tobacco raw material having a low content of specified polycyclic aromatic hydrocarbons and low-molecular carboxylic acids and containing a large amount of specified flavor components, and a method for manufacturing the tobacco raw material. The objective is achieved with a manufacturing method including: a step for drying pre-deribbing leaf tobacco after har-

vesting until the moisture content thereof reaches 10-25 wt%; a step for smoke-processing the pre-deribbing dried tobacco using thermally decomposed smoke at 400-500°C; and a step for, after the smoke-processing step, storing the pre-deribbing smoke-processed leaf tobacco under partially sealed conditions.

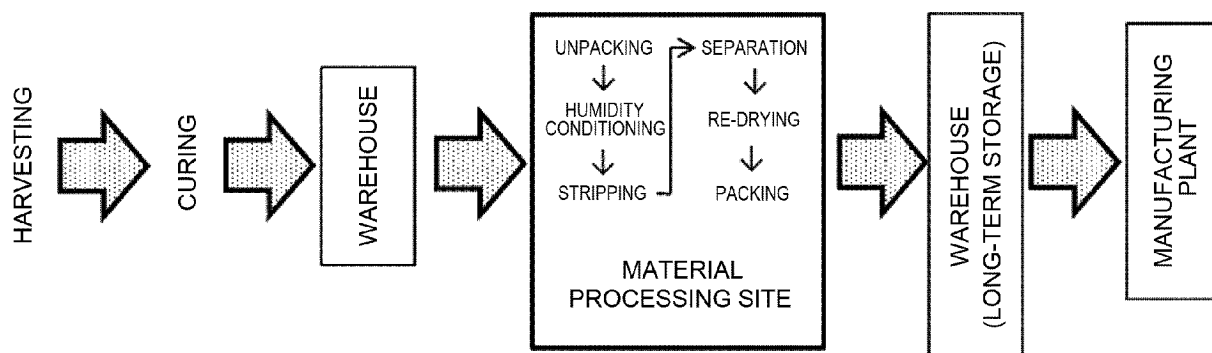


FIG. 1

Description

[Technical Field]

5 **[0001]** The present invention relates to a method for manufacturing a tobacco material and to a tobacco material.

[Background Art]

10 **[0002]** Efforts have already been made to improve the flavor of leaf tobacco as a raw material. For example, Non Patent document 1 describes passing smoke from rice husks or wood through shredded tobacco to impart a distinctive smoky aroma and describes the generation of smoke in this case by gently heating rice husks to gradually effect charring and burning.

15 **[0003]** Dark fire-cured (DFC) is known as a tobacco material that has undergone a smoke treatment. The general manufacturing method here is known to include a step of subjecting a raw tobacco leaf that has not undergone a drying treatment to a smoke treatment with smoke generated by the combustion of, e.g., wood, at a heating/combustion temperature of approximately 600°C to 900°C; a subsequent fermentation step; and an ensuing storage step after going through an ageing step.

[0004] DFC is used as a starting material for, e.g., snuff, pipe tobacco, and chewing tobacco.

20 **[0005]** Patent document 1 describes a tobacco product that uses an extract of a fire-cured tobacco and states that the content of the benzo[a]pyrene contained in ordinary fire-cured tobacco is approximately 150 to approximately 800 ng/g.

[0006] Patent document 2, meanwhile, describes the production of ester compounds as brought about by the addition of an alcohol such as ethyl alcohol after the execution of a smoke treatment on a dried leaf tobacco, followed by storage of the alcohol-added leaf tobacco to bring about an esterification reaction between organic acids provided by the smoke treatment and the subsequently added alcohol.

25 [Citation List]

[Patent document]

30 **[0007]**

[Patent document 1] Japanese Translation of PCT Application No. 2013-542744

[Patent document 2] WO 2014/203341

35 [Non Patent document]

[0008] [Non Patent document 1] Tobacco Academic Studies Center, The Encyclopedia of Tobacco, Japan, San-ai Shoin (Yugen Kaisha (Y.K.)), March 31, 2009, First Edition, page 327

40 [Summary of Invention]

[Technical Problem]

45 **[0009]** In Non Patent document 1, only ordinary smoking means and so forth are described as the methods for imparting a distinctive smoky aroma to a tobacco material such as shredded tobacco.

[0010] In the invention described in Patent document 2, the smoke treatment is carried out on the dried leaf tobacco in order to provide the dried leaf tobacco with substrates for the subsequent esterification reaction by providing the cured leaf tobacco with the organic acids present in the smoke, and the object of this invention is simply to increase the ester compounds in the tobacco material.

50 **[0011]** In contrast to this, the present invention addresses the problem of providing a tobacco material that exhibits a suppression of the adhesion to the leaf tobacco of the benzo[a]pyrene that can be produced in the case of execution of the ordinary smoke treatments as heretofore carried out, that exhibits a reduced content of low molecular weight carboxylic acids, and that exhibits an increase in specific flavor components, and of providing a method for manufacturing this tobacco material.

55 [Solution to Problem]

[0012] As a result of intensive investigations by the present inventor, it was discovered that a tobacco material having

a low content of benzo[a]pyrene, a low content of low molecular weight carboxylic acids, and a large content of specific flavor components, can be provided by a manufacturing method containing a step of drying a post-harvest, unstripped leaf tobacco to a moisture content of 10 to 25 wt%; a step of subjecting the dried unstripped leaf tobacco to a smoke treatment with a smoke provided by the pyrolysis of a smoke-producing material in a material temperature range of 400°C to 500°C; and a step, subsequent to the smoke treatment step, of storing the smoke-treated unstripped leaf tobacco under a semi-sealed condition. The present invention was achieved based on this discovery.

[0013] That is, embodiments of the present invention are as follows.

[1] A method for manufacturing a tobacco material, including the steps of: drying a post-harvest unstripped leaf tobacco to a moisture content of 10 to 25 wt%; subjecting the dried unstripped leaf tobacco to a smoke treatment with a smoke provided by pyrolysis of a smoke-producing material in a material temperature range of 400°C to 500°C; and, subsequent to the smoke treatment step, storing the smoke-treated unstripped leaf tobacco under a semi-sealed condition.

[2] The method for manufacturing a tobacco material according to [1], wherein the drying of the post-harvest unstripped leaf tobacco is carried out to a moisture content thereof of 15 to 25 wt%.

[3] The method for manufacturing a tobacco material according to [1] or [2], wherein the storing step is carried out at room temperature for a duration of at least 3 months but less than 24 months.

[4] The method for manufacturing a tobacco material according to any of [1] to [3], wherein the tobacco material yielded by the manufacturing method has the following constitution:

- (1) a benzo[a]pyrene content is not more than 100 ng/g based on the dry weight of the tobacco material;
- (2) a acetic acid content is not more than 20 mg/g based on the dry weight of the tobacco material;
- (3) a guaiacol content is 5 to 5,000 μ g/g based on the dry weight of the tobacco material; and
- (4) a 2,6-dimethoxyphenol content is 10 to 10,000 μ g/g based on the dry weight of the tobacco material.

[5] A tobacco material constituted of a leaf tobacco that has not been subjected to a lamina/stem separation process, wherein the tobacco material has a constitution according to the following (1) to (4):

- (1) a benzo[a]pyrene content is not more than 100 ng/g based on the dry weight of the tobacco material;
- (2) an acetic acid content is not more than 20 mg/g based on the dry weight of the tobacco material;
- (3) a guaiacol content is 5 to 5,000 μ g/g based on the dry weight of the tobacco material; and
- (4) a 2,6-dimethoxyphenol content is 10 to 10,000 μ g/g based on the dry weight of the tobacco material.

[6] A tobacco product comprising the tobacco material according to [5].

[7] The tobacco product according to [6], which is a cigarette or a heating flavor inhaler.

[Advantageous Effects of Invention]

[0014] The present invention thus provides a tobacco material having a low content of benzo[a]pyrene, a low content of low molecular weight carboxylic acids, and a high content of specific flavor components, and provides a method for manufacturing this tobacco material.

[Brief Description of Drawings]

[0015]

Fig. 1 is a schematic diagram that shows a general process for manufacturing a tobacco product.

Fig. 2 is a diagram that shows the relationship between the material temperature of a smoke-producing material and the content of the benzo[a]pyrene contained in a leaf tobacco after a smoke treatment.

Fig. 3 is a diagram that shows the relationship between the material temperature of a smoke-producing material and the content of the guaiacol contained in a leaf tobacco after a smoke treatment.

Fig. 4 is a diagram that shows the relationship between the material temperature of a smoke-producing material and the content of the 2,6-dimethoxyphenol contained in a leaf tobacco after a smoke treatment.

Fig. 5 is a schematic diagram of an apparatus used to measure the adhesion efficiency by specific flavor components to a leaf tobacco.

Fig. 6 is a diagram that shows the relationship between the moisture content of a pre-smoke-treatment leaf tobacco and the guaiacol adhesion efficiency.

Fig. 7 is a diagram that shows the relationship between the moisture content of a pre-smoke-treatment leaf tobacco

and the 2,6-dimethoxyphenol adhesion efficiency.

Fig. 8 is a diagram that shows the timewise change in the propionic acid content in leaf tobacco during storage under a semi-sealed condition and under a sealed condition.

Fig. 9 is a diagram that shows the timewise change in the acetic acid content in leaf tobacco during storage under a semi-sealed condition and under a sealed condition.

Fig. 10 is a diagram that shows the timewise change in the propionic acid content in leaf tobacco during storage (accelerated test) under a semi-sealed condition.

Fig. 11 is a diagram that shows the timewise change in the acetic acid content in leaf tobacco during storage (accelerated test) under a semi-sealed condition.

[Description of Embodiments]

[0016] The present invention is described in detail in the following using embodiments, examples, and so forth. However, the present invention is not limited to or by the following embodiments, examples, and so forth, and can be executed using any variation or modification within a range in which there is no departure from the essential features of the present invention.

[0017] Fig. 1 shows a common manufacturing process for a smoking article, e.g., cigarettes and so forth.

[0018] In this manufacturing process, the harvested leaf tobacco is stored in a warehouse after undergoing a curing treatment.

[0019] This curing is a treatment that is carried out in a first stage on the harvested leaf tobacco, and it generally includes a step of drying, humidity conditioning, and so forth and may also include activation of the action of various enzymes present in leaf tobacco.

[0020] Curing is performed mainly for the following purposes:

(1) increasing the action of enzymes and causing the development of changes in the leaf color; (2) drying the leaf lamina and fixing the leaf color; and (3) removing the moisture in the stem and drying out the leaf as a whole.

[0021] After this curing, storage in a warehouse is carried out for a prescribed period of time followed by transport of the leaf tobacco to a material processing site. Stripping of the leaf tobacco and separation are not carried out during curing, and these processes, e.g., stripping and so forth, are performed at the material processing site.

[0022] The manufacturing method according to embodiments of the present invention is carried out in place of this curing treatment, and the tobacco material provided by proceeding through the manufacturing method according to embodiments of the present invention is generally transported to the material processing site after warehouse storage.

[0023] In contrast to this, the manufacturing method described in Patent document 2 is carried out on a tobacco material that has undergone the treatments at the material processing site shown in Fig. 1. The reason for this is as follows: when the treatments commonly performed at the material processing site as shown in Fig. 1 are carried out on the tobacco material provided by the manufacturing method described in Patent document 2, the ester compounds present in the tobacco material end up vaporizing due to the performance of treatments that are accompanied by heating, e.g., re-drying and so forth. This is also supported by the fact that the leaf tobacco treated by the manufacturing method described in Patent document 2 is a "shredded tobacco", for which the assumption is made of having been processed at the material processing site of Fig. 1.

[0024] Based on the preceding, the manufacturing method described in Patent document 2 and the manufacturing method according to embodiments of the present invention thus have completely different circumstances of execution.

[0025] In this Description, "leaf tobacco" indicates leaf tobacco prior to the execution, or in the course of the execution, of the process according to the steps in the manufacturing method according to the present invention, while "tobacco material" designates the material that has been processed.

<Step of Drying Post-harvest Unstripped Leaf Tobacco>

[0026] The post-harvest unstripped leaf tobacco used for the tobacco material according to embodiments of the present invention is leaf tobacco after the "harvesting" shown in Fig. 1, but prior to the occurrence of a color change such as yellowing, prior to the execution of the "curing" treatment, and prior to stripping.

[0027] There are no particular limitations on the leaf tobacco used in the manufacturing method according to embodiments of the present invention or used for the tobacco material described in the following, and examples here are genus *Nicotiana* and burley varieties, flue-cured varieties, Oriental varieties, and domestic varieties of *Nicotiana tabacum* and *Brasilia* varieties of *Nicotiana rustica*.

[0028] With the regard to the post-harvest leaf tobacco, the use is particularly preferred of the leaf tobacco immediately after harvesting, and the drying treatment described below is preferably carried out on such leaf tobacco immediately after harvesting.

[0029] The post-harvest leaf tobacco (raw leaf) ordinarily has a high moisture content and has a moisture content of

80 to 90 wt% with reference to the total amount of the leaf tobacco.

[0030] A drying treatment is carried out on this leaf tobacco until the moisture content reaches 10 to 25 wt%.

[0031] The lower limit for the moisture content of the leaf tobacco provided by the drying treatment is more preferably 12 wt% and particularly preferably is 15 wt%. When the range for the moisture content of the leaf tobacco is 15 to 25 wt%, a particularly excellent effect is obtained with regard to increasing the content (adhesion amount) of the specific flavor components that are provided by the smoke. The upper limit on the moisture content of the leaf tobacco can also be, for example, 20 wt%.

[0032] The specific flavor components can be exemplified by guaiacol and 2,6-dimethoxyphenol.

[0033] The moisture content in the leaf tobacco is determined using the following method.

[0034] Based on a method for analyzing the moisture in foods (thermal drying method), heating is carried out for 1 hour at 100°C under normal pressure followed by spontaneous cooling in a desiccator for 40 minutes. The moisture is determined from the weight difference pre-versus-post-heating. The specific procedure is as follows.

(1) The empty weight of a sample container stored in the desiccator is measured.

(2) The necessary amount of the leaf tobacco is weighed out and introduced into the sample container and the lid is applied.

(3) The lid of the sample container is removed, the sample container is placed in a rotary drying vessel, and heating is carried out for 1 hour at 100°C.

(4) After 1 hour, the sample container is closed with the lid and is removed and spontaneous cooling is carried out in a desiccator.

(5) After 40 minutes, the weight of the sample container is measured. The moisture is determined using the following formula.

$$Mw = \frac{W1-W2}{W1-W0} \times 100$$

Mw: moisture (%)

W1: weight of the sample container and pre-dried sample (g)

W2: weight of the sample container and dried sample (g)

W0: weight of the sample container (g)

[0035] There are no particular limitations on the drying treatment method, and the following methods can be provided as examples.

[0036] In one method, the leaf tobacco is air-dried for approximately 1 month at approximately 20°C to 50°C, and preferably 25°C to 35°C, although this will vary depending on the region where the tobacco grows, in a drying room that is impermeable to direct sunlight, e.g., a pipe-house, by suitable gapless row drying or stem drying such that drying unevenness cannot occur, while the air is undergoing natural convection.

[0037] In another method, drying is carried out for approximately 5 days at a temperature of approximately 30°C to 70°C and a relative humidity of approximately 20% to 100% in an apparatus such as a circulation bulk dryer that supports temperature and humidity control.

[0038] These two methods may also be used in combination.

[0039] In another method, drying is carried out for several hours with an apparatus at a drying temperature of approximately 100°C in the apparatus compartment.

<Step of Performing Smoke Treatment>

[0040] In the step of subjecting the leaf tobacco to a smoke treatment, a smoke-producing material is heated so as to provide a material temperature for the smoke-producing material, e.g., wood, of 400°C to 500°C in order to produce smoke as described in the following.

[0041] By heating to a material temperature for the smoke-producing material in the range from 400°C to 500°C, in contrast to the DFC manufacturing method described above, specific polycyclic aromatic hydrocarbons, for example, benzo[a]pyrene, which are adhered in large amounts to the leaf tobacco with DFC, can be reduced. On the other hand, by heating the smoke-producing material so as to reach a material temperature of at least 400°C, the pyrolysis of substances, for example, lignins, present in the smoke-producing material is promoted and specific flavor components, and specifically guaiacol and 2,6-dimethoxyphenol, can be adhered to the leaf tobacco in satisfactory amounts.

[0042] The smoke-producing material can be exemplified by wood, and the type thereof is not particularly limited and can be, for example, white oak, cherry, walnut, apple, beech, oak, and hickory.

[0043] The duration of the smoke treatment can be adjusted as appropriate, for example, in the range from approximately 10 minutes to 6 months.

[0044] All of these smoke treatments can be carried out using a known smoking device, smoking apparatus, and so forth. The smoke treatment can be carried out in a state where the leaf tobacco is stacked or the leaf tobacco can be row dried or stem dried.

[0045] In the manufacturing method according to embodiments of the present invention, it is unnecessary to perform any treatment and/or to add any substance after the aforementioned smoke treatment step and before the storage step described below in order to modify the properties of the leaf tobacco or the substances imparted by the smoke treatment. Such a treatment and such a substance addition are ordinarily not performed.

[0046] For example, the DFC manufacturing method described above includes a fermentation step and an ageing step after performing the smoke treatment and before the storage step, whereas the method according to the present invention do not include such a fermentation step and an aging step.

[0047] In addition, for example, the step of adding an alcohol in order to produce ester compounds in a subsequent step, as described in Patent document 2, is also not included in the manufacturing method according to the present invention.

[0048] Accordingly, the manufacturing method according to embodiments of the present invention contains the storage step described in the following after the step of subjecting the leaf tobacco to a smoke treatment and following this smoke treatment step.

<Storage Step>

[0049] In order to volatilize low molecular weight carboxylic acids, e.g., acetic acid, during storage, the step of storing the smoke-treated unstripped leaf tobacco is carried out under a semi-sealed condition.

[0050] This semi-sealed condition can be, for example, a condition in which the smoke-treated leaf tobacco is inserted into an air-permeable packing material, such as a vinyl bag, and the opening of this packing material is fold over, thereby impeding the inflow and outflow of air to a certain degree.

[0051] The storage of the leaf tobacco under semi-sealed conditions can be achieved by filling the packing material as described above with the smoke-treated leaf tobacco and folding the opening over to produce a leaf tobacco-filled, semi-sealed packing material, and storing this leaf tobacco-filled packing material in a storage container, e.g., a cardboard carton.

[0052] In this storage, the storage of a large amount of the leaf tobacco at a single time may be carried out by producing a plurality of the leaf tobacco-filled packing materials and storing same stacked within a storage container.

[0053] By carrying out the storage step under semi-sealed conditions in the present invention, the content of low molecular weight carboxylic acids in the resulting tobacco material can be reduced because low molecular weight carboxylic acids such as acetic acid, which can cause irritation, then undergo volatilization during storage.

[0054] The duration of storage should be a time interval that supports a satisfactory reduction in the low molecular weight carboxylic acids such as acetic acid, but is not otherwise particularly limited, although at least 3 months is preferred. The upper limit on the duration of storage, on the other hand, is preferably approximately 24 months considering the time interval until the produced tobacco material is shipped to the material processing site.

[0055] The temperature during storage is preferably a temperature at which the low molecular weight carboxylic acids undergo a satisfactory volatilization.

[0056] The specific temperature can be exemplified by room temperature. The specific temperature range can be exemplified by the range of 5°C to 60°C, and the temperature can also be adjusted as appropriate in the range from 10°C to 55°C.

[0057] An additive, e.g., an essential oil, aroma extract, and so forth, may be added in a freely selected amount in the manufacturing method according to the present invention in any step except between the smoke treatment step and the storage step.

[0058] A step of adjusting the moisture content of the obtained tobacco material may be present after the storage step. By going through such a step, the moisture content of the tobacco material can be adjusted to 10 to 20 wt% and preferably 10 to 15 wt% with reference to the total amount of the tobacco material.

[0059] As indicated in the preceding, the manufacturing method according to embodiments of the present invention is performed prior to the execution of the treatments at the tobacco material processing site as shown in Fig. 1. The tobacco material yielded by the manufacturing method according to the present invention is ordinarily stored in a warehouse and then transported to the tobacco material processing site (refer to Fig. 1). This storage in a warehouse is ordinarily for approximately 10 to 180 days.

[0060] A tobacco material having the same component content as the <Tobacco Material> described in the following can be obtained in accordance with the method for manufacturing a tobacco material that has been described in the preceding.

<Tobacco Material>

[0061] The tobacco material according to embodiments of the present invention, for example, can be manufactured by the method according to the present invention as described above for manufacturing a tobacco material.

[0062] Leaf tobacco constituting the tobacco material according to embodiments of the present invention is leaf tobacco prior to the execution of stripping at the material processing site shown in Fig. 1, and is constituted of leaf tobacco that has not been subjected to a lamina/stem separation process (is unthreshed).

[0063] The types of leaf tobacco constituting the tobacco material according to embodiments of the present invention may be the same types as used in the manufacturing method described above.

[0064] The tobacco material according to embodiments of the present invention has a constitution according to the following (1) to (4):

(1) the benzo[a]pyrene content is not more than 100 ng/g based on the dry weight of the tobacco material;

(2) the acetic acid content is not more than 20 mg/g based on the dry weight of the tobacco material;

(3) the guaiacol content is 5 to 5,000 $\mu\text{g/g}$ based on the dry weight of the tobacco material; and

(4) the 2,6-dimethoxyphenol content is 10 to 10,000 $\mu\text{g/g}$ based on the dry weight of the tobacco material.

[0065] In addition, the benzo[a]pyrene content of the tobacco material according to embodiments of the present invention is more preferably not more than 50 ng/g on a dry weight basis and is particularly preferably not greater than the detection limit. A low amount of adhesion by harmful substances contained in the smoke is provided by having the benzo[a]pyrene content be in this range.

[0066] When a tobacco material is produced by the manufacturing method described in the preceding, the benzo[a]pyrene content in the tobacco material according to embodiments of the present invention can be adjusted by adjusting the material temperature of the smoke-producing material when the smoke treatment is performed. For example, when a wood is used for the smoke-producing material provided to the aforementioned smoke treatment, the content of specific polycyclic aromatic hydrocarbon can be limited into the range indicated above when the material temperature of the wood is adjusted into the range from 400°C to 500°C.

[0067] In addition, the tobacco material according to embodiments of the present invention preferably has an acetic acid content of not more than 20 mg/g on a dry weight basis and particularly preferably not more than 15 mg/g on a dry weight basis.

[0068] A tobacco material exhibiting little irritation for the user is provided by having the acetic acid content be in the indicated range. The tobacco material according to the present invention, on the other hand, may contain acetic acid at or above 10 $\mu\text{g/g}$.

[0069] The propionic acid content in the tobacco material according to embodiments of the present invention is preferably not more than 1 mg/g on a dry weight basis and is particularly preferably not more than 0.5 mg/g on a dry weight basis. In another embodiment, the tobacco material according to the present invention can contain propionic acid at or above 10 $\mu\text{g/g}$.

[0070] When the tobacco material is produced by the manufacturing method described above, the acetic acid content and propionic acid content in the tobacco material according to embodiments of the present invention can be adjusted by adjusting the storage temperature and storage time. For example, when the storage time is extended, the amount of volatilized low molecular weight carboxylic acid is increased, and as a consequence the content of low molecular weight carboxylic acids in the tobacco material can be further reduced.

[0071] The tobacco material according to embodiments of the present invention can be manufactured by the above-described manufacturing method according to embodiments of the present invention; however, the manufacturing method according to embodiments of the present invention does not contain the step of adding an alcohol in order to produce ester compounds that is described in Patent document 2. As a consequence, the tobacco material according to embodiments of the present invention substantially does not contain the ester compounds described in Patent document 2, for example, ethyl acetate and ethyl valerate. Here, "substantially does not contain" indicates at or below the detection limit.

[0072] The guaiacol content in the tobacco material according to the present invention is more preferably 5 to 5,000 $\mu\text{g/g}$ on a dry weight basis and is particularly preferably 100 to 1,500 $\mu\text{g/g}$ on a dry weight basis.

[0073] The 2,6-dimethoxyphenol content in the tobacco material according to the present invention is more preferably 10 to 10,000 $\mu\text{g/g}$ on a dry weight basis and is particularly preferably 500 to 2,000 $\mu\text{g/g}$ on a dry weight basis.

[0074] Guaiacol and 2,6-dimethoxyphenol are components specific to the smoke and are components that provide the user with smokiness, an aroma and taste characteristic of tobacco materials, and a flavor with an impact on the perception of migrating from the oral cavity to the nasal cavity.

[0075] When the tobacco material is produced by the above-described manufacturing method, the content of the guaiacol and 2,6-dimethoxyphenol in the tobacco material according to embodiments of the present invention can be adjusted by adjusting the duration of the smoke treatment. For example, the duration of the smoke treatment is extended

when a greater addition of these components is sought.

[0076] The ratio between the 2,6-dimethoxyphenol content and the guaiacol content (also referred to as the 2,6-DMP/G ratio in the following) in the tobacco material according to embodiments of the present invention is preferably 1 to 20, more preferably 2 to 15, and particularly preferably 2 to 10.

[0077] Having the 2,6-DMP/G ratio be in the indicated range makes it possible to provide the user with a good balance of smokiness, an aroma and taste characteristic of tobacco materials, and an impact on the perception of migrating from the oral cavity to the nasal cavity.

[0078] This 2,6-DMP/G ratio can be adjusted by changing, for example, the smoking conditions (type of wood chip, pyrolysis temperature, and so forth) and the storage conditions (temperature, extent of contact with air, duration, and so forth).

[0079] For example, the 2,6-DMP/G ratio declines when the material temperature of the smoke-producing material in the smoke treatment step is raised; the 2,6-DMP/G ratio increases when the temperature that is a condition of storage is raised. In addition, the 2,6-DMP/G ratio increases when the extent of contact with air that is a storage condition is increased. The 2,6-DMP/G ratio also increases when the duration of storage is extended.

[0080] In conventional DFC, because a drying treatment is not carried out prior to the smoke treatment, the moisture content of the leaf tobacco is substantially higher than for the leaf tobacco according to the present invention and the amount of adhesion by 2,6-dimethoxyphenol to the leaf tobacco is smaller. In addition, in conventional DFC, the material temperature of the smoke-producing material in the smoke treatment is higher than in the present invention and as a consequence 2,6-DMP/G assumes a declining trend. Accordingly, the 2,6-DMP/G ratio in conventional DFC is presumed to be smaller than the 1 that is the lower limit on the range indicated above.

[0081] The ratio between the 2,6-dimethoxyphenol content and the phenol content (also referred to as the 2,6-DMP/P ratio) in the tobacco material according to embodiments of the present invention is preferably 1 to 100, more preferably 5 to 70, and particularly preferably 20 to 50.

[0082] The smoky sensation is made more prominent by having the 2,6-DMP/P ratio be in the indicated range.

[0083] This 2,6-DMP/P ratio, for example, is reduced when the material temperature of the smoke-producing material in the smoke treatment step is increased, and is reduced when the duration of storage that is a condition of storage is extended. It is also reduced when the temperature during storage is increased.

[0084] In conventional DFC, because a drying treatment is not carried out prior to the smoke treatment, the moisture content of the leaf tobacco is substantially higher than in the leaf tobacco according to the present invention and the amount of adhesion by 2,6-dimethoxyphenol to the leaf tobacco is smaller. Accordingly, the 2,6-DMP/P ratio in conventional DFC is presumed to be smaller than the 1 that is the lower limit on the range indicated above.

[0085] The ratio between the guaiacol content and the phenol content (also referred to as the G/P ratio) in the tobacco material according to embodiments of the present invention is preferably 0.5 to 12.0, more preferably 0.5 to 6.0, and particularly preferably 2.0 to 5.0.

[0086] The smoky sensation is made prominent by having the G/P ratio be in the indicated range.

[0087] This G/P ratio, for example, is reduced when the material temperature of the smoke-producing material in the smoke treatment step is increased, and the G/P ratio is reduced when the temperature that is a condition of storage is increased.

[0088] The moisture content for the tobacco material according to embodiments of the present invention can be, for example, 10 to 20 wt% in an embodiment and is preferably 10 to 15 wt%.

[0089] The content of polycyclic aromatic hydrocarbon (benzo[a]pyrene) in the tobacco material can be determined by the following method.

[0090] 0.5 g of dried shredded tobacco is accurately weighed into a glass screw-cap vial (capacity = 20 mL); 200 μ L of internal reference substance (deuterated form of the particular polycyclic aromatic hydrocarbon) and 10 mL of cyclohexane as extraction solvent are added; the cap is applied; and extraction is carried out by shaking at 190 rpm for 30 minutes at room temperature. The supernatant is filtered across a 0.45- μ m membrane filter and is subsequently concentrated to 1 mL, purified and concentrated on a solid-phase extraction SPE column (SPE: Si 2 g/12 cc, small amount of Na₂SO₄), and then dispensed into a GC vial. Analysis is carried out with a GCMS equipped with an autosampler, and the amount of each target component in the sample is quantitated by the internal reference method.

[0091] The following conditions can be used for the GC/MS conditions.

<GC/MS Analysis Conditions>

[0092] Instrument: gas chromatography analysis instrument (5975N) from Agilent Technologies, Inc.

[0093] Injection port conditions:

Injection port temperature: 300°C; Injection method: pulsed splitless; and Injection amount: 1 μ L

Analysis column: DB-17 ms from Agilent Technologies, Inc., 30 m × 0.25 mm × 0.25 μm

Column flow rate: 1.2 mL/min. (He)

Temperature ramp conditions:

Initial: 60°C (1 min); then 15°C/min to 150°C (0.5 min); then 5°C/min to 320°C (18.5 min); then Run time (60 min.)

MS conditions:

Ion source: 300°C;

Quadrupole: 180°C;

SIM mode

[0094] The contents of the low molecular weight carboxylic acids in the tobacco material, starting with acetic acid, and the contents of the flavor components (guaiacol, 2,6-dimethoxyphenol, phenol) in the tobacco material can be determined by the following method.

[0095] 0.5 g of the dried and ground tobacco material is accurately weighed into a glass screw-cap vial (capacity = 20 mL) and 0.05 μg quinoline is added as an internal reference substance. 10 mL of methanol is added as extraction solvent; the cap is applied; and extraction is carried out by shaking at 200 rpm for 60 minutes at room temperature. The supernatant is filtered across a 0.45-μm membrane filter and is then transferred into a GC vial; analysis is carried out with a GC/MS equipped with an autosampler. The amount of the analysis target component contained in each sample is quantitated by determining the ratio between the peak areas for the analysis target component and quinoline on the obtained GC chromatogram.

[0096] The following conditions can be used for the GC/MS conditions.

Instrument: gas chromatography analysis instrument (6890N) from Agilent Technologies, Inc., and mass detector (5973N) from Agilent Technologies, Inc.

Amount injected: 1 μL (injection in pulsed splitless mode)

Column: HP-INNOWAX (30 m × 0.25 mm (0.25 μm film thickness)) from Agilent Technologies, Inc.

Oven: 40°C → 260°C (5°C/min)

Mass detector: TIC mode (mass numbers 29 to 550)

<Tobacco Product>

[0097] The tobacco material according to embodiments of the present invention is a unstripped tobacco material in which the lamina and stems remain present as such.

[0098] Accordingly, when the tobacco material according to embodiments of the present invention is used to produce a tobacco product as exemplified hereinafter, it can be used as a starting material for the tobacco product by going through a stripping step and separation step at the material processing site as shown in Fig. 1. Thus, in another embodiment, the hereabove-described method for manufacturing a tobacco material includes stripping and separation steps after the step of storing the smoke-treated unstripped leaf tobacco under semi-sealed conditions.

[0099] The starting material shipped from the material processing site shown in Fig. 1 may be blended at a manufacturing plant in any proportions with, for example, an ordinary tobacco material, and a cigarette may be produced using this blended starting material.

[0100] In addition, the tobacco material according to embodiments of the present invention may be disposed in a filter portion of a known cigarette, for example, in a form of a blend in any proportions with an ordinary shredded tobacco. The disposition in the filter portion may be, for example, a disposition within a cavity or may be a disposition dispersed in the filter fiber.

[0101] The tobacco material may also be used in a heating flavor inhaler or in an unheated flavor inhaler.

[0102] When the tobacco material according to embodiments of the present invention is used in a cigarette or heating flavor inhaler, the user may then enjoy the flavor generated by the specific flavor components described above.

[0103] A "heating flavor inhaler" is an inhaler in which the tobacco material is heated without combustion and the user experiences the flavor of the heated tobacco material by inhalation. Examples are a carbon heat source-type inhaler in which a tobacco material is heated by the combustion heat of a carbon heat source (refer, for example, to WO 2006/073065), an electrically heated inhaler provided with an inhaler and a heating device for electrically heating the inhaler (refer, for example, to WO 2010/110226), and a liquid atomization inhaler in which a liquid aerosol source containing a tobacco material is atomized by heating (refer, for example, to WO 2015/046385).

[0104] Among these, the electrically heated flavor inhaler contains, inter alia, a mouthpiece, a main unit containing an electronic heater and a container holding a composition that contains a tobacco material, and a temperature controller for controlling the temperature of the electronic heater. The specific structure described in Japanese Translation of PCT Application No. 2014-524313 can be used. The container holding the tobacco material-containing composition can be, for example, a pod.

[0105] The tobacco material according to embodiments of the present invention may be contained the above-mentioned container, for example, in a form of a blend in any proportions with the ordinary shredded tobacco. The material of a container to contain the tobacco material according to embodiments of the present invention is not particularly limited, and examples thereof include a metal with high thermal conductivity such as aluminum.

[0106] When used in the aforementioned heating flavor inhaler, the tobacco material according to embodiments of the present invention may be stored in the container in the form of a composition containing, for example, a blend of the tobacco material according to embodiments of the present invention in any proportions with, for example, an ordinary shredded tobacco, and also containing a polyhydric alcohol such as glycerol and/or propylene glycol, a thickener, and other optional components such as a fragrance.

[0107] In a preferred embodiment, the composition used in a heating flavor inhaler contains the tobacco material according to embodiments of the present invention, a polyhydric alcohol, and a thickener.

[0108] The size of each leaf tobacco present in the tobacco material is also not particularly limited, and the sizes used in common electrically heated flavor inhalers can be used.

[0109] The weight proportion of the tobacco material according to embodiments of the present invention in the composition filled in the container can be, for example, approximately 10% to 40%.

[0110] In the case of liquid atomization inhalers in which a liquid aerosol source containing a tobacco material is atomized by heating, the tobacco material according to embodiments of the present invention may be used as a portion of the starting material for producing the liquid flavor source inserted in the liquid reservoir.

[0111] Snus is an example of the use of the tobacco material according to embodiments of the present invention as an oral tobacco product. In this case, production is carried out by filling, using a known method, a blend in any proportions of a tobacco material produced by the above-described manufacturing method with, for example, a common shredded tobacco, into a packaging material that uses a starting material such as, for example, a nonwoven fabric. For example, the tobacco material in an adjusted amount may be filled and sealed by any means such as heat sealing to obtain snus.

[0112] There are no particular limitations on the packing material that may be used, but, for example, a cellulosic nonwoven fabric is preferably used.

[0113] When the oral tobacco product is, for example, a gum, production is carried by blending, using a known method, a known gum base with the above-described tobacco material obtained using the manufacturing method according to embodiments of the present invention. Also with regard to chewing tobacco, pipe tobacco, and compressed tobacco, production can be carried out using known methods, but using the above-described tobacco material obtained using the manufacturing method according to embodiments of the present invention. Also with regard to edible films, production can be carried out using known materials and methods, but using the above-described tobacco material obtained using the manufacturing method according to embodiments of the present invention.

[0114] The manufacturing method according to embodiments of the present invention solves, for example, the problem of the adhesion to leaf tobacco of polycyclic aromatic hydrocarbon present in smoke produced at a temperature at which the occurrence of incomplete combustion is facilitated, a point that has been a problem for tobacco material subjected to a conventional smoke treatment such as DFC, and solves the problem of the production of irritating substances such as acetic acid, while providing a tobacco material in which components exhibiting the characteristic flavor of smoke are increased.

[0115] The tobacco material manufactured with the method according to the present invention may be used as a portion of the tobacco material in tobacco products. The tobacco material yielded by the present invention, when used as a portion of the tobacco material in a tobacco product, may be used in any proportion.

Examples

[0116] The present invention is more specifically described using examples, but the present invention is not limited to the description in the following examples as long as the essential features of the present invention are not exceeded.

<Experimental Example 1>

Leaf Tobacco Drying Treatment

[0117] Immediately after harvesting, leaf tobacco was air-dried in a drying room impermeable to direct sunlight, by row drying or stem drying, for approximately 1 month in the temperature range from 25°C to 35°C.

[0118] The post-drying moisture content of the leaf tobacco was adjusted to be 5 to 50 wt% for each sample.

Smoke Treatment of Leaf Tobacco

[0119] A Compact Smoker (Snow Peak Inc.) was used for the smoking apparatus. Approximately 20 g of wood chips

was placed in the bottom; approximately 10 g of the unstripped leaf tobacco, dried as described above, was stacked on the upper level; and the lid was applied. This was placed, with the bottom of the smoking apparatus grounded, on a temperature-controllable heating apparatus (AS ONE Corporation, Hot Plate CHR-250DN). The set temperature of the heating apparatus was set to 500°C and the wood chips were pyrolyzed. Ten minutes was used for the pyrolysis time of the wood chips, and white oak, walnut, hickory, oak, or walnut was used as the smoke-producing material.

[0120] The material temperature of the smoke-producing material was adjusted to be approximately from 300°C to 600°C.

Storage Treatment of Leaf Tobacco

[0121] The smoke-treated material was introduced into a zip-equipped vinyl bag (Seisannipponsha Ltd.) and Lamizip AL-E (Seisannipponsha Ltd.). A semi-sealed system was set up by closing the top of the zip-equipped vinyl bag into a lightly collapsed configuration. The top of the Lamizip AL-E, on the other hand, was heat sealed to set up a completely sealed system. These bags were stored in a thermostatted room at a temperature of 22°C and a humidity of 62%. The duration of storage was up to 4 months.

<Differences in Content of Benzo[a]pyrene, Guaiacol, and 2,6-Dimethoxyphenol Based on Differences in Temperature of Smoke Treatment of Leaf Tobacco>

[0122] The benzo[a]pyrene content, guaiacol content, and 2,6-dimethoxyphenol content were each measured on the leaf tobacco provided by carrying out the smoke treatment at a material temperature for the smoke-producing material of 300°C, 400°C, 450°C, 500°C, or 600°C on the leaf tobacco provided by the drying treatment described in Experimental Example 1 (moisture content: 13 wt%). For reference, the contents were measured for the original raw material, i.e., the untreated leaf tobacco, and the contents were also measured for conventional DFC (execution of a smoke treatment on the tobacco leaf hung as such in a state without separation of the stems from the lamina).

(1) Benzo[a]pyrene

[0123] The results of the benzo[a]pyrene measurements are given in Fig. 2.

[0124] As is clear from Fig. 2, the content of benzo[a]pyrene was low at 300°C to 500°C, while the benzo[a]pyrene content was dramatically increased at 600°C. This shows that benzo[a]pyrene is present at high contents in leaf tobacco yielded by the execution of a smoke treatment at a temperature in excess of 600°C, as in conventional DFC.

[0125] For comparison, the benzo[a]pyrene content in the original raw material is also given in Fig. 2.

[0126] The benzo[a]pyrene contents given in Fig. 2 are collected in the following Table 1.

[Table 1]

material temperature of smoke-producing material (°C)	BaP (ng/g-DB)
300	4.4
400	5.1
450	5.4
500	4.8
600	26.2
* BaP content of original raw material: 3.6 ng/g-DB, BaP content for conventional DFC: 600.5 ng/g-DB	

(2) Guaiacol

[0127] The results of the guaiacol measurements are given in Fig. 3.

[0128] As is clear from Fig. 3, it was demonstrated that guaiacol was contained in the leaf tobacco in large amounts when the material temperature of the smoke-producing material was 400°C to 500°C, and that guaiacol was contained in the largest amount at 450°C. The content of guaiacol contained in the original raw material and the guaiacol content for conventional DFC are also shown in Fig. 3 for comparison. As shown in Fig. 3, almost no guaiacol is present in the original raw material. This is because guaiacol is produced by the pyrolysis of lignin and is provided to the leaf tobacco by smoke treatment.

(3) 2,6-Dimethoxyphenol

[0129] The measurement results for 2,6-dimethoxyphenol are shown in Fig. 4.

[0130] As is clear from Fig. 4, it was demonstrated that 2,6-dimethoxyphenol was contained in the leaf tobacco in large amounts when the material temperature of the smoke-producing material was 400°C to 500°C. The content of 2,6-dimethoxyphenol contained in the original raw material and the 2,6-dimethoxyphenol content for conventional DFC are also shown in Fig. 4 for comparison.

[0131] As shown in Fig. 4, almost no 2,6-dimethoxyphenol is present in the original raw material. This is because 2,6-dimethoxyphenol is produced by the pyrolysis of lignin and is provided to the leaf tobacco by smoke treatment.

[0132] The guaiacol content and 2,6-dimethoxyphenol content are collected in the following Table 2.

[Table 2]

material temperature of smoke-producing material (°C)	guaiacol (μg/g-DB)	2,6-dimethoxyphenol (μg/g-DB)
300	32.0	828.3
400	137.6	2672.4
450	228.1	2654.1
500	152.0	2406.4
600	88.0	1334.3
* Original raw material: guaiacol content = 1.3 μg/g-DB, 2,6-dimethoxyphenol content = 8.4 μg/g-DB		
* Conventional DFC: guaiacol content = 52.7 μg/g-DB, 2,6-dimethoxyphenol content = 1043.1 μg/g-DB		

<Relationship between Moisture Content of Leaf Tobacco Prior to Smoke Treatment and Guaiacol Adhesion Efficiency and 2,6-Dimethoxyphenol Adhesion Efficiency>

[0133] In order to elucidate the relationship between the moisture content of the leaf tobacco after the drying treatment (before the smoke treatment) in Experimental Example 1 and the adhesion efficiency to the leaf tobacco of guaiacol and 2,6-dimethoxyphenol due to the smoke treatment, tests were carried out using the apparatus shown in Fig. 5 instead of the smoke treatment described for Experimental Example 1.

[0134] In the apparatus in Fig. 5, leaf tobacco 3 that has undergone the drying treatment in Experimental Example 1 is placed in the center of the interior of the cylinder and a filter 4 is placed on the downstream side of the air flow therefrom. A smoke treatment was performed by heating the smoke-producing material 1 using a hot plate 2, and the following were measured: the amount of guaiacol and the amount of 2,6-dimethoxyphenol adhered to the leaf tobacco 3 and the amount of guaiacol and the amount of 2,6-dimethoxyphenol adhered to the filter 4.

[0135] The adhesion efficiencies were calculated using the following formula.

$$\text{adhesion efficiency (\%)} = \frac{\text{amount of guaiacol or 2,6-dimethoxyphenol adhered to the leaf tobacco}}{\text{amount of guaiacol or 2,6-dimethoxyphenol bound to the leaf tobacco} + \text{amount of guaiacol or 2,6-dimethoxyphenol adhered to the filter}} \times 100$$

[0136] For guaiacol and 2,6-dimethoxyphenol, plots are given in Figs. 6 and 7, respectively, in which the moisture content after the drying treatment (before the smoke treatment) is plotted on the horizontal axis and the adhesion efficiency is plotted on the vertical axis.

Results for Adhesion Efficiency

[0137] The adhesion efficiency for both guaiacol and 2,6-dimethoxyphenol was increased when the moisture content

of the leaf tobacco after the drying treatment (before the smoke treatment) was 15 to 25 wt%. A moisture content for the leaf tobacco after the drying treatment in excess of 30 wt% is unfavorable because fermentation due to the action of microorganisms present in the leaf tobacco may then advance and the balance among the components of the leaf tobacco may be disrupted.

<Timewise Variation in Contents of Acetic Acid and Propionic Acid in Leaf Tobacco during Storage under Semi-sealed and Sealed Conditions>

[0138] The post-smoke-treatment leaf tobacco provided by Experimental Example 1 was stored in a semi-sealed system and in a sealed system, and the acetic acid and propionic acid contents were measured when 0, 1, 2, 3, and 4 months had elapsed for the duration of storage. The temperature during storage was 22°C at 66% RH. The moisture content of the leaf tobacco upon storage was 15 wt%.

[0139] The results for propionic acid are given in Fig. 8, and the results for acetic acid are given in Fig. 9.

[0140] According to the results in Figs. 8 and 9, the propionic acid and acetic acid contents for storage in a semi-sealed system were reduced from those for storage in a sealed system with greater elapsed time and particularly when the duration of storage reached 4 months.

<Timewise Variation in Contents of Propionic Acid and Acetic Acid in Leaf Tobacco during Storage under Semi-sealed Conditions (Accelerated Testing)>

[0141] The post-smoke-treatment leaf tobacco provided by Experimental Example 1 was submitted to storage in a semi-sealed system using accelerated testing (40°C, 60% RH). Four months in this accelerated testing is hypothesized to correspond to approximately 24 months of testing at 22°C. The results for propionic acid are given in Fig. 10, and the results for acetic acid are given in Fig. 11.

[0142] The results in Figs. 10 and 11 demonstrated that the propionic acid content and acetic acid content in the stored leaf tobacco underwent an ongoing decline as the duration of storage in the semi-sealed system grew longer.

[Industrial Applicability]

[0143] The manufacturing method according to the present invention can provide a tobacco material that has a low benzo[a]pyrene content and a low content of low molecular weight carboxylic acids such as acetic acid and propionic acid and that contains large amounts of specific flavor components.

Claims

1. A method for manufacturing a tobacco material, comprising the steps of:

drying a post-harvest unstripped leaf tobacco to a moisture content of 10 to 25 wt%;
subjecting the dried unstripped leaf tobacco to a smoke treatment with a smoke provided by pyrolysis of a smoke-producing material in a material temperature range of 400°C to 500°C; and,
subsequent to the smoke treatment step, storing the smoke-treated unstripped leaf tobacco under a semi-sealed condition.

2. The method for manufacturing a tobacco material according to claim 1, wherein the drying of the post-harvest unstripped leaf tobacco is carried out to a moisture content thereof of 15 to 20 wt%.

3. The method for manufacturing a tobacco material according to claim 1 or 2, wherein the storing step is carried out at room temperature for a duration of at least 3 months but less than 24 months.

4. The method for manufacturing a tobacco material according to any one of claims 1 to 3, wherein the tobacco material yielded by the manufacturing method has a constitution according to the following (1) to (4):

- (1) a benzo[a]pyrene content is not more than 100 ng/g based on the dry weight of the tobacco material;
- (2) an acetic acid content is not more than 20 mg/g based on the dry weight of the tobacco material;
- (3) a guaiacol content is 5 to 5,000 μ g/g based on the dry weight of the tobacco material; and
- (4) a 2,6-dimethoxyphenol content is 10 to 10,000 μ g/g based on the dry weight of the tobacco material.

5. A tobacco material constituted of a leaf tobacco that has not been subjected to a lamina/stem separation process, wherein the tobacco material has a constitution according to the following (1) to (4):

- (1) a benzo[a]pyrene content is not more than 100 ng/g based on the dry weight of the tobacco material;
(2) an acetic acid content is not more than 20 mg/g based on the dry weight of the tobacco material;
(3) a guaiacol content is 5 to 5,000 $\mu\text{g/g}$ based on the dry weight of the tobacco material; and
(4) a 2,6-dimethoxyphenol content is 10 to 10,000 $\mu\text{g/g}$ based on the dry weight of the tobacco material.

6. A tobacco product comprising the tobacco material according to claim 5.

7. The tobacco product according to claim 6, which is a cigarette or a heating flavor inhaler.

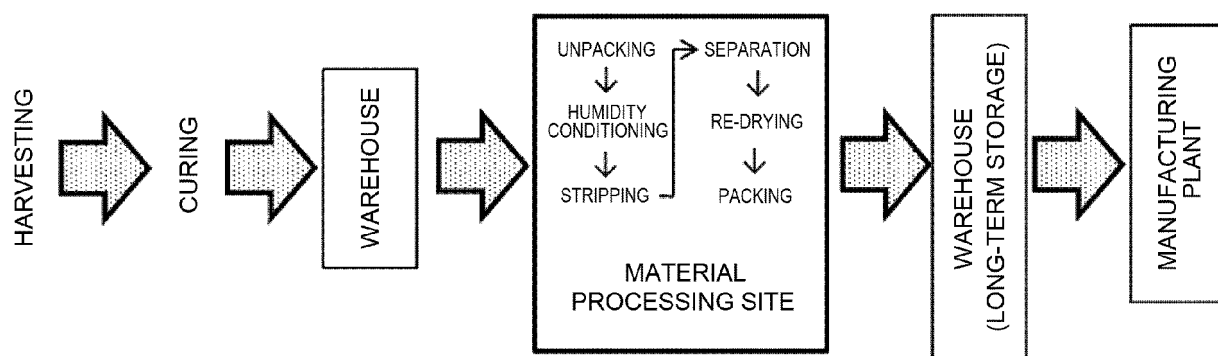


FIG. 1

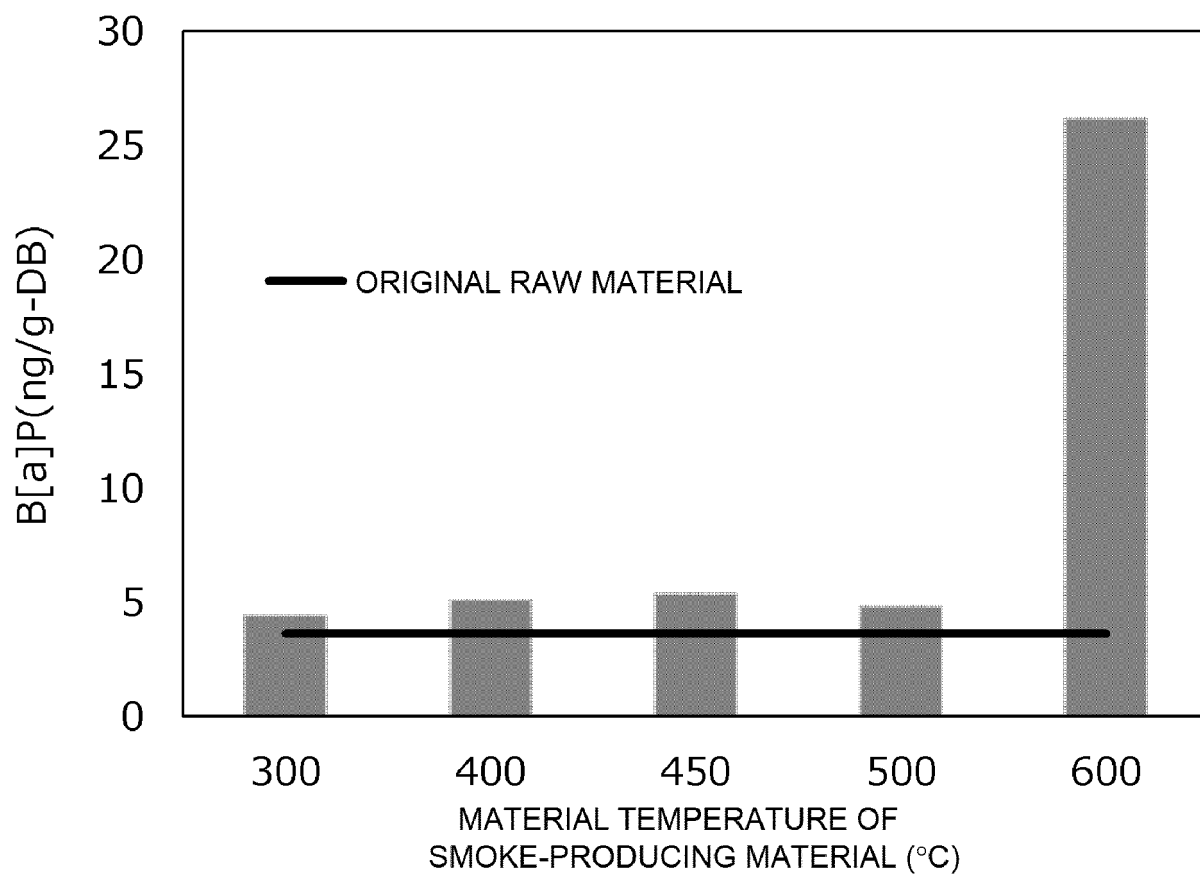


FIG. 2

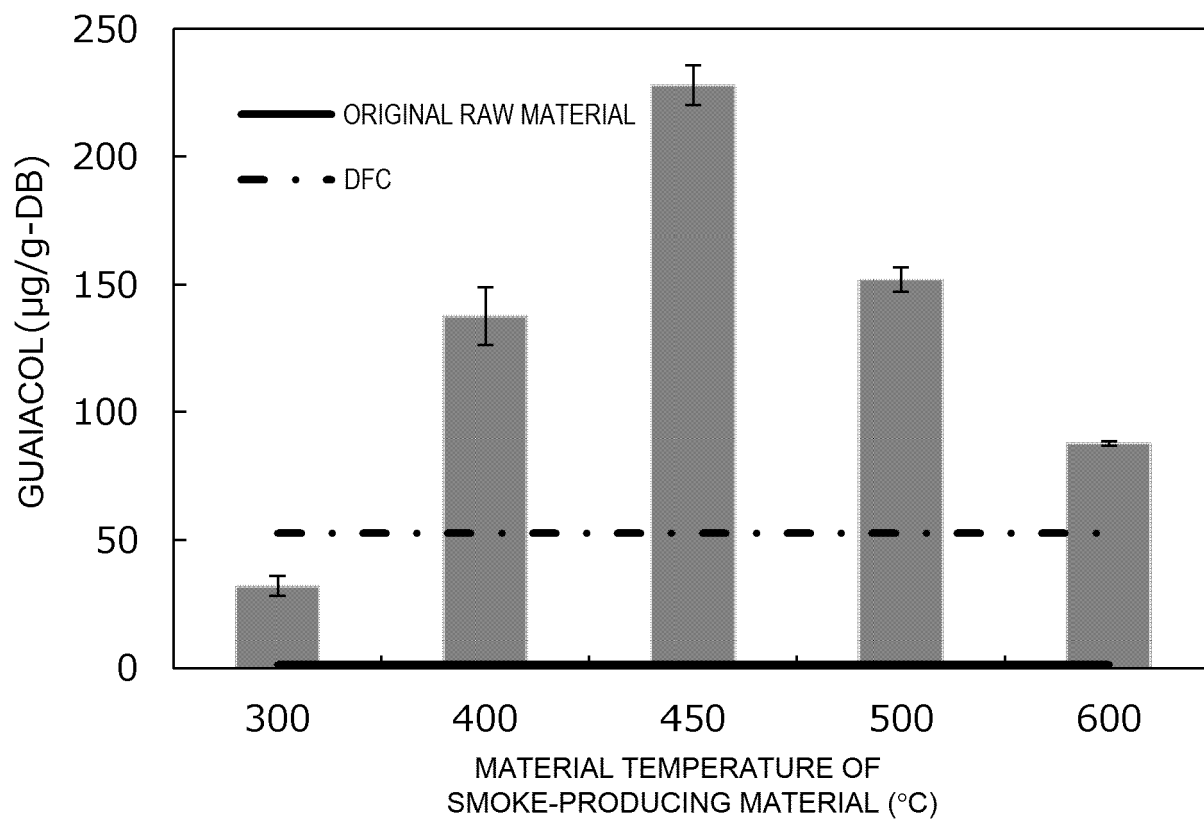


FIG. 3

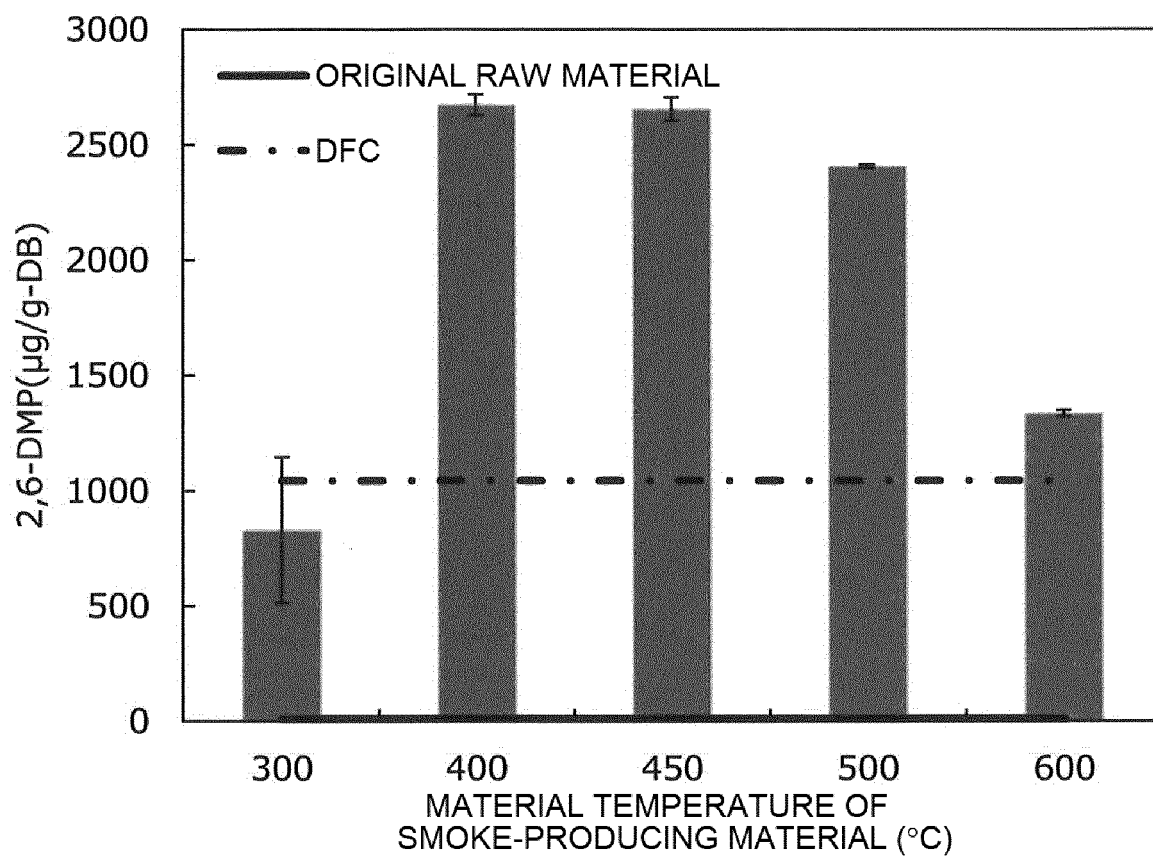


FIG. 4

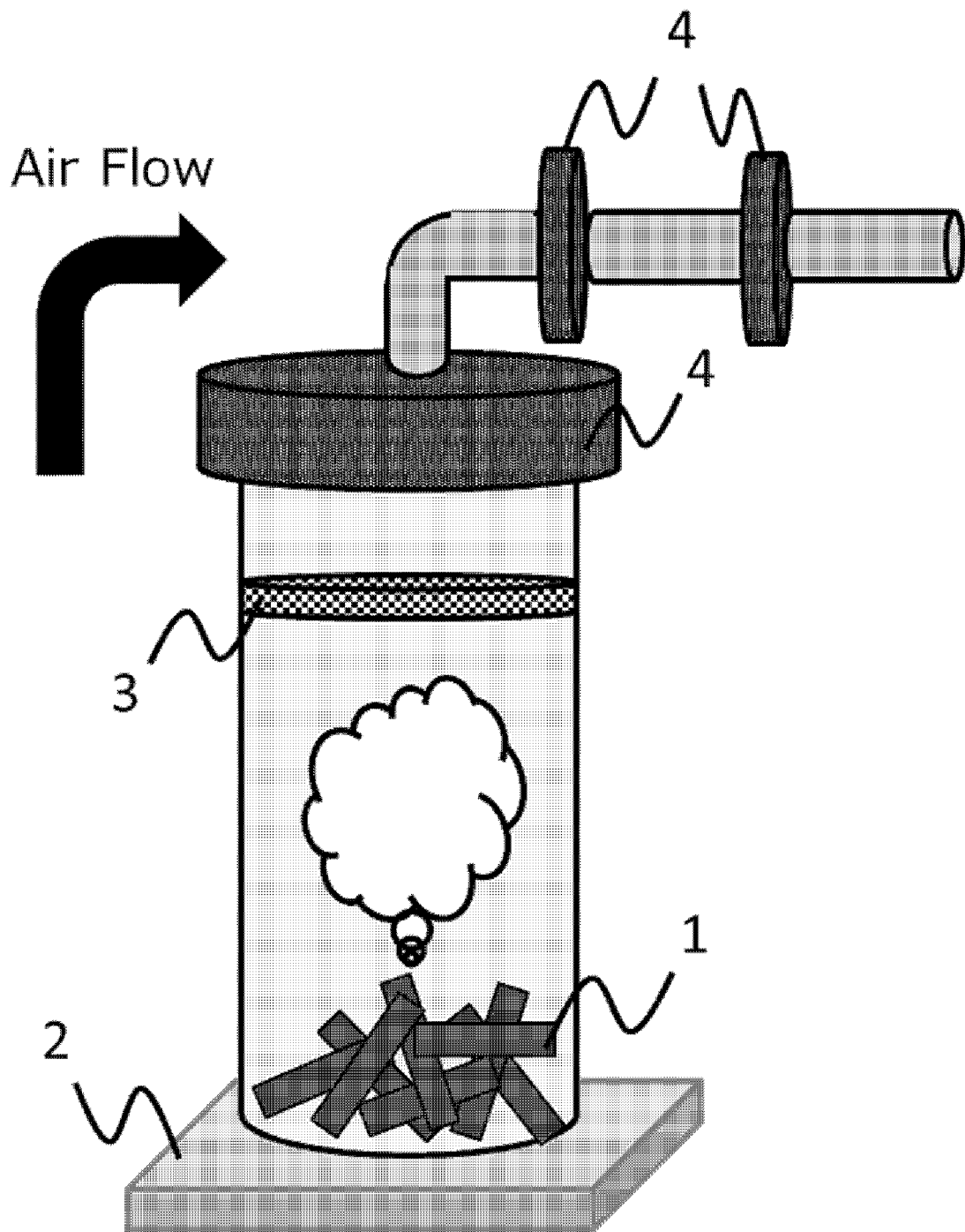


FIG. 5

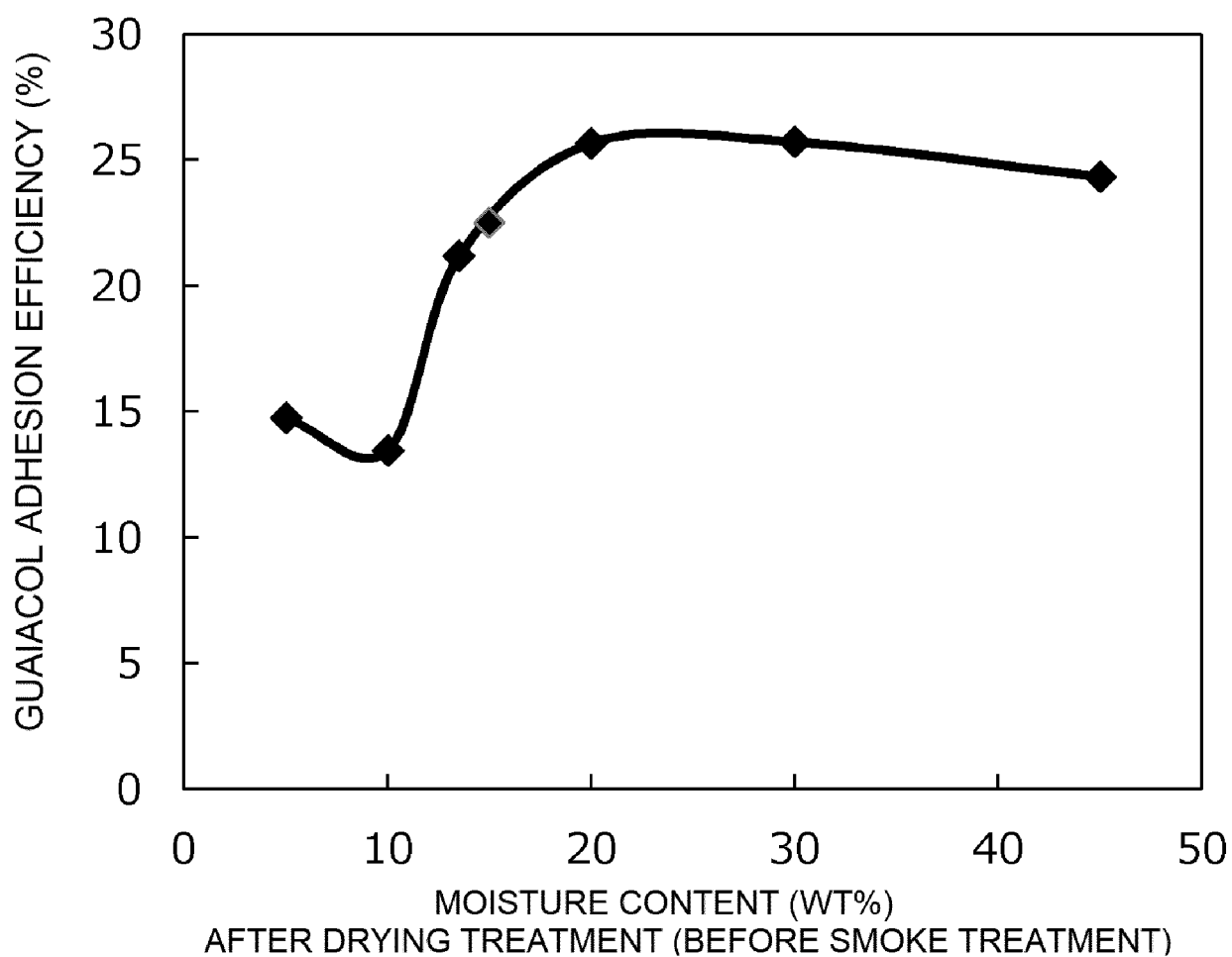


FIG. 6

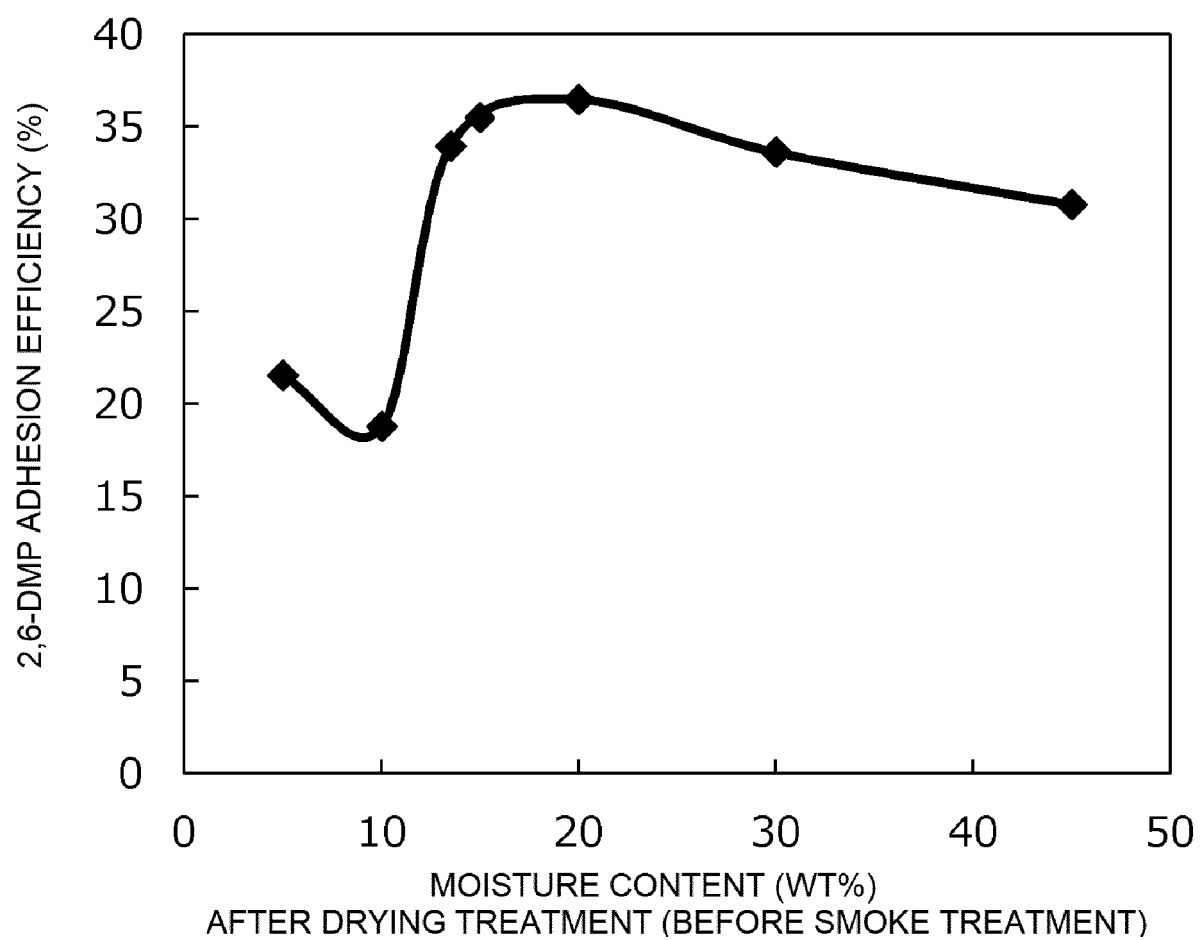


FIG. 7

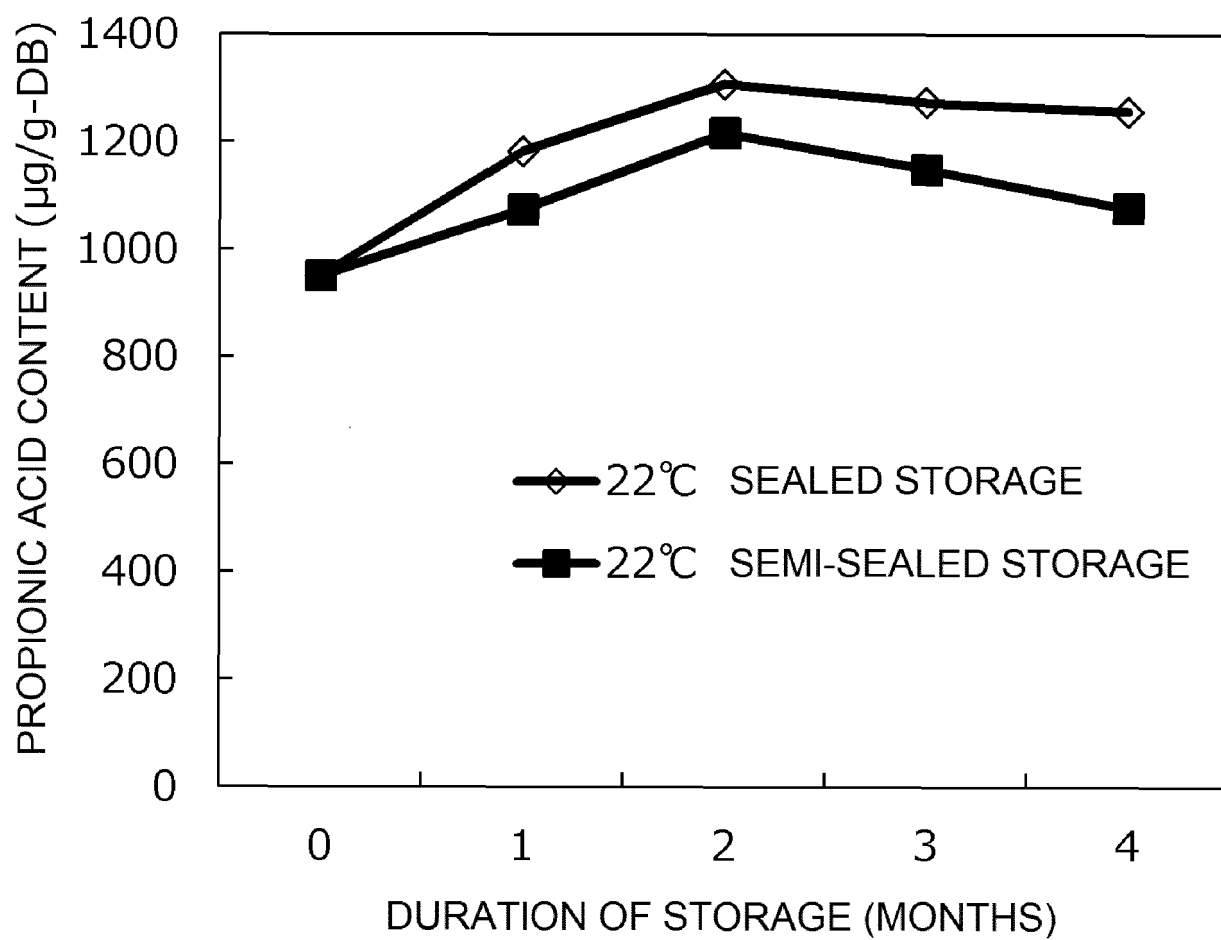


FIG. 8

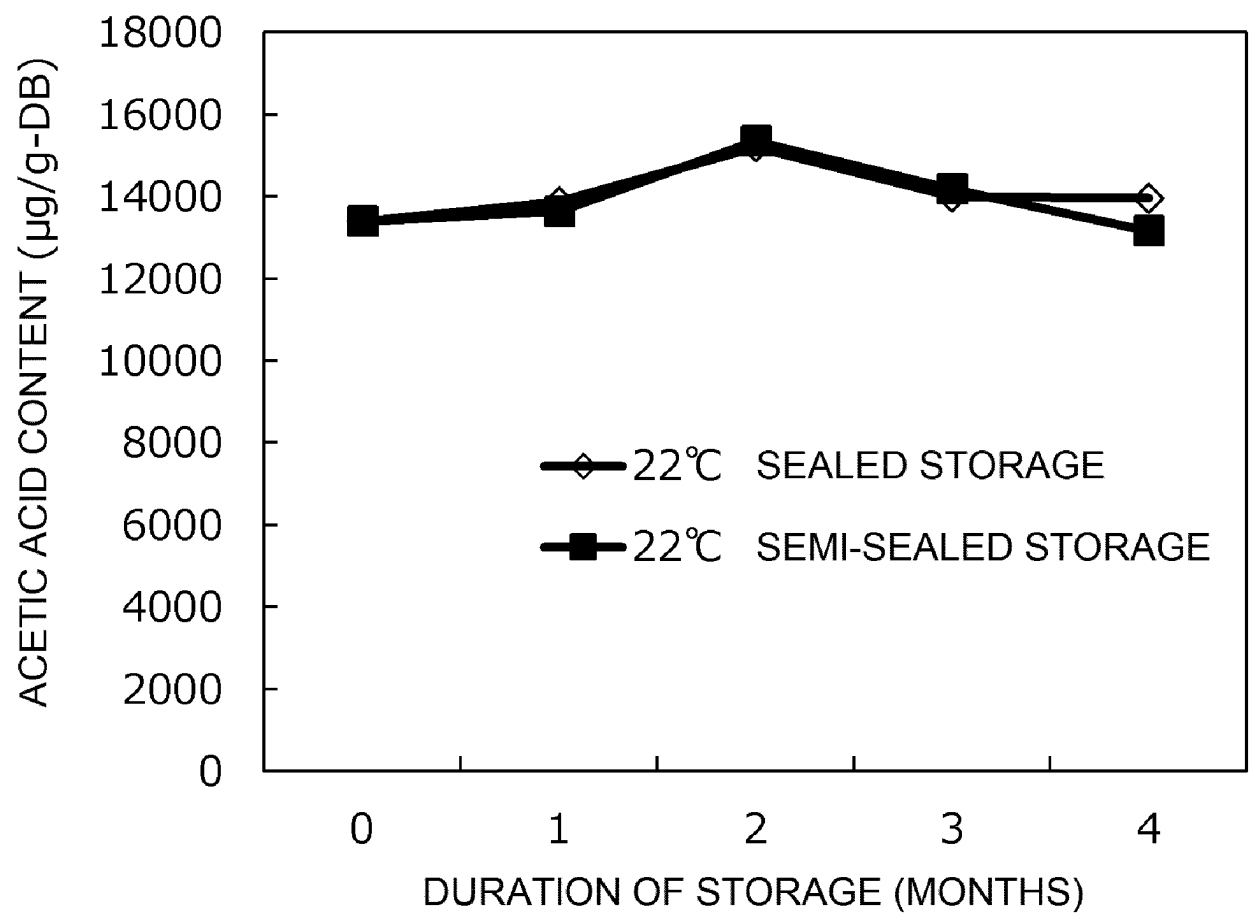


FIG. 9

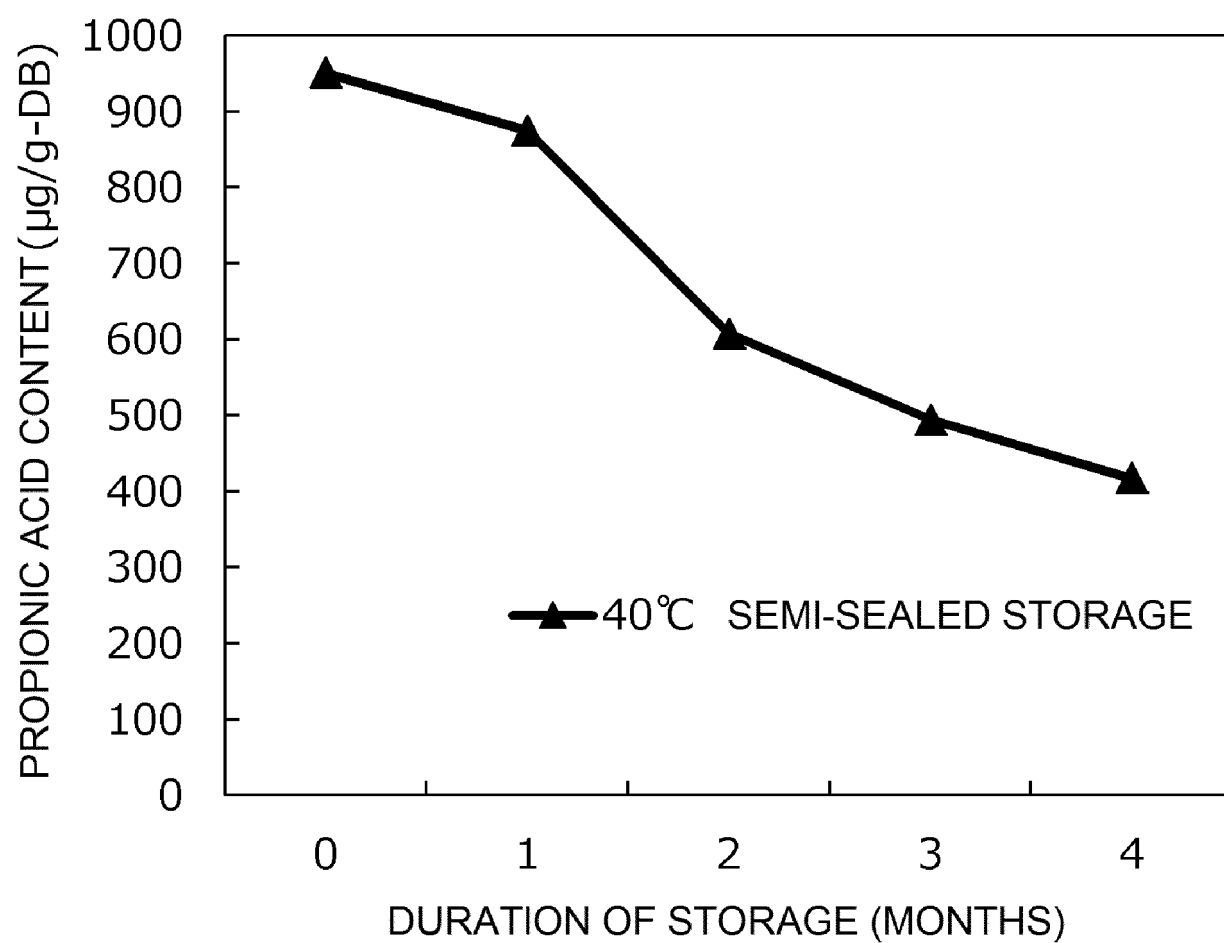


FIG. 10

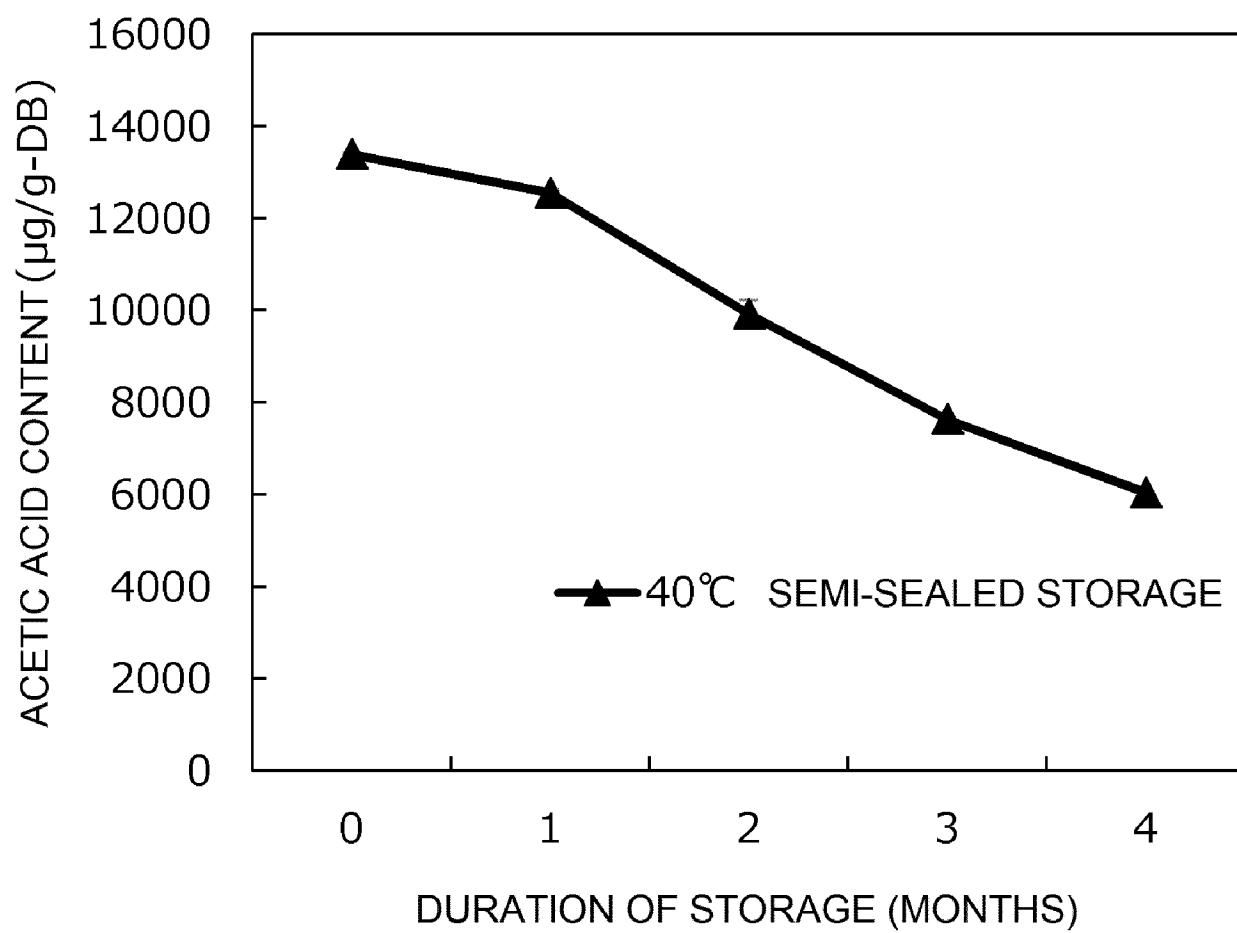


FIG. 11

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2017/044045

A. CLASSIFICATION OF SUBJECT MATTER
Int. Cl. A24B3/12 (2006.01) i

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)
Int. Cl. A24B3/12

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 1971-2017

Registered utility model specifications of Japan 1996-2017

Published registered utility model applications of Japan 1994-2017

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.
Y	WO 2014/203341 A1 (JAPAN TOBACCO INC.) 24 December 2014, paragraph [0013], claims 1, 3, 4 & EP 2965637 A1, paragraphs [0032]-[0038], claims 1, 3, 4 & CN 105307515 A	1-4
Y	JP 2016-526921 A (R.J. REYNOLDS TOBACCO COMPANY) 08 September 2016, claims 5, 22-29, paragraph [0105] & US 2015/0040922 A1, claims 5, 22-29, paragraph [0074] & WO 2015/021137 A1 & EP 3030098 A & CN 105530824 A	1-7



Further documents are listed in the continuation of Box C.



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Date of the actual completion of the international search

Date of mailing of the international search report

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Authorized officer

Telephone No.

Form PCT/ISA/210 (second sheet) (January 2015)

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2017/044045

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 2013-542744 A (R.J. REYNOLDS TOBACCO COMPANY) 28 November 2013, claim 5 & US 2012/0125354 A1, claim 5 & US 2012/0199145 A1 & WO 2012/068375 A1 & WO 2013/142483 A1 & EP 2640204 A & EP 2827727 A & CN 103369978 A & ES 2515716 T & CN 104284605 A	4-7

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REFERENCES CITED IN THE DESCRIPTION

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- WO 2014203341 A [0007]
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- WO 2010110226 A [0103]
- WO 2015046385 A [0103]
- JP 2014524313 W [0104]

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