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(71) Applicant: Japan Tobacco Inc.

Tokyo 105-6927 (JP)

(72) Inventors:

 YAMAUCHI, Shun Tokyo 130-8603 (JP)

 KOBAYASHI, Shunzo Tokyo 130-8603 (JP)

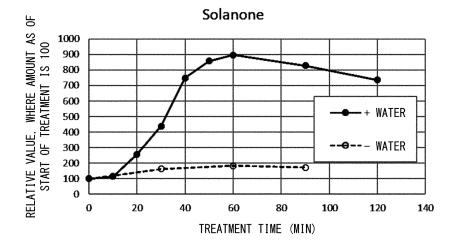
(74) Representative: Hoffmann Eitle
Patent- und Rechtsanwälte PartmbB
Arabellastraße 30
81925 München (DE)

(54) METHOD FOR AUGMENTING AMOUNT OF SOLANONE IN LEAF TOBACCO, METHOD FOR MANUFACTURING TOBACCO RAW MATERIAL, AND METHOD FOR EXTRACTING SOLANONE

(57) Provided is a technique for increasing an amount of solanone in a tobacco leaf. A method for increasing an amount of solanone in a tobacco leaf in accordance with an embodiment of the present invention

includes a heating step of heating a tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water.





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#### **Description**

Technical Field

**[0001]** The present invention relates to a method for increasing an amount of solanone in a tobacco leaf, a method for producing a tobacco material, and a method for extracting solanone.

**Background Art** 

[0002] Solanone is a type of unsaturated ketone and is one of aroma components contained in a tobacco plant. Solanone contained in a tobacco leaf is known to have effects such as improving a mouth-feel of tobacco smoke. [0003] In order to increase an amount of solanone in a tobacco leaf, studies have been conducted with focus on a curing technique. Non-patent Literature 1 discloses such a method, specifically, a method of air curing a tobacco leaf under conditions ranging from 20°C in temperature and 90% in humidity to 35°C in temperature and 70% in humidity.

Citation List

[Non-patent Literatures]

[0004] [Non-patent Literature 1]

IIDA, Bunkichi et al. Changes in Chemical Composition of Burley Variety during Curing (Part 2) Factors in Production of Solanone and Norsolanadione. Tobacco Leaf Study. 1997, vol. 132, pp. 79-89

Summary of Invention

**Technical Problem** 

**[0005]** However, a method capable of further increasing an amount of solanone in a tobacco leaf is yet to be established. A technique for increasing an amount of solanone in a tobacco leaf is therefore still awaited.

**[0006]** It is an object of an aspect of the present invention to provide a technique for increasing an amount of solanone in a tobacco leaf.

Solution to Problem

**[0007]** The inventors of the present invention found, through diligent study, that heating a tobacco leaf under predetermined conditions makes it possible to increase an amount of solanone in the tobacco leaf, and consequently completed the present invention.

**[0008]** In order to attain the object, a method for increasing an amount of solanone in a tobacco leaf in accordance with an aspect of the present invention includes a heating step of heating a tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water.

**[0009]** A method for producing a tobacco material in accordance with an aspect of the present invention includes: a heating step of heating a tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water; and a curing step of subjecting, to a curing treatment, the tobacco leaf which has been subjected to the heating step.

**[0010]** A method for extracting solanone in accordance with an aspect of the present invention includes: a heating step of heating a tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water; and an extraction step of extracting solanone from the tobacco leaf which has been subjected to the heating step.

Advantageous Effects of Invention

**[0011]** According to an aspect of the present invention, it is possible to increase an amount of solanone in a tobacco leaf.

**Brief Description of Drawings** 

[0012]

Fig. 1 is a diagram showing amounts of solanone contained in cured leaves which have been heated for various heat treatment times.

Fig. 2 is a diagram showing amounts of solanone contained in heated fresh leaves of various tobacco varieties.

Fig. 3 is a diagram showing amounts of solanone contained in cured leaves which have been heated at various temperatures.

Fig. 4 is a diagram showing amounts of solanone contained in cured leaves which have been heated by various heating methods.

Fig. 5 is a diagram showing amounts of  $\beta$ -damascenone and 3-oxo- $\alpha$ -ionol contained in a cured leaf which has been heated.

Description of Embodiments

[1. Method for increasing amount of solanone in tobacco leaf]

[0013] A method for increasing an amount of solanone in a tobacco leaf in accordance with an aspect of the present invention (hereinafter, simply referred to as "increasing method") includes a heating step of heating a tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water. This configuration makes it possible to increase an amount of solanone contained in a tobacco leaf.

<Increase in amount of solanone in tobacco leaf>

[0014] In the present specification, an "increase in

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amount of solanone in a tobacco leaf" means that an amount of solanone is preferably not less than two times, more preferably not less than three times, even more preferably not less than four times, and particularly preferably not less than five times that of an amount of solanone in a tobacco leaf that has not been subjected to a heating step. An increase in amount of solanone can be confirmed by a known method that can quantify solanone. For example, solanone in a tobacco leaf can be extracted with use of an organic solvent such as hexane, and an amount of the solanone can be measured with use of a gas chromatography mass spectrometer.

#### <Heating step>

**[0015]** The heating step is a step of heating a tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water.

**[0016]** In the increasing method in accordance with an aspect of the present invention, it is only necessary that the tobacco leaf be a leaf harvested from a tobacco plant, and the tobacco plant is not limited to a specific type. However, the tobacco plant is preferably *Nicotiana tabacum*. Further, the tobacco plant can be of any variety such as (i) a cultivar such as flue-cured variety or Burley variety or (ii) a local variety.

[0017] The state of a tobacco leaf to be heated is not particularly limited. For example, the tobacco leaf can be a fresh leaf, or can be a cured leaf. The tobacco leaf can also be a leaf that has been stored for a predetermined period, an aged leaf, or the like. In a case where the tobacco leaf is a fresh leaf, it is sufficient to carry out curing only once after carrying out the increasing method in accordance with an aspect of the present invention. This makes it possible to save time and cost. Thus, from the viewpoint of reducing cost, the tobacco leaf is preferably a fresh leaf. From the viewpoint of preservability, the tobacco leaf is preferably a cured leaf. In the present specification, a "fresh leaf" means a leaf that has not been cured after being harvested from a tobacco plant, and a "cured leaf" means a leaf that has been cured after being harvested from a tobacco plant. In a case where a tobacco leaf is cured, stored, or aged, the curing, storing, or aging can be carried out by a known method.

**[0018]** The form of a tobacco leaf to be heated is not particularly limited. Examples of the form of a tobacco leaf include cut fillers, powder, sheets, stems, and granules as well as the tobacco leaf itself. A tobacco leaf can be made into such a form by a known method.

**[0019]** In the present specification, "in the presence of water" means any state in which sufficient water is present on the surface of a tobacco leaf. Examples of such a state include a state in which steam is sufficiently in contact with the surface of a tobacco leaf, a state in which the surface of a tobacco leaf is wet, and a state in which a tobacco leaf is present in water. Such a state can be achieve, for example, by a method such as: immersing a tobacco leaf in water; adding water to a tobacco leaf;

putting a tobacco leaf in a space filled with steam obtained by boiling water; or (i) immersing a tobacco leaf in water or adding water to a tobacco leaf and (ii) putting the tobacco leaf in a space filled with steam obtained by boiling water. The form of the water can be steam, liquid, or a state in which steam and a liquid coexist. From the viewpoint of preventing an escape of a component in a tobacco leaf, the water is preferably steam, and a state in which the steam is sufficiently in contact with the surface of the tobacco leaf is preferable. Hereinafter, a tobacco leaf that has been subjected to the heating step and is in a state where water is present on the surface of the tobacco leaf is also referred to as a "tobacco leaf sample".

[0020] A tobacco leaf sample can have a water content of not less than 50%, not less than 60%, not less than 70%, not less than 80%, or not less than 90%. In the present specification, a "water content of a tobacco leaf sample" means a total of (ia) an amount of water contained in the tobacco leaf and (ii) a weight of water used in immersion of the tobacco leaf, water added to the tobacco leaf, or water adhering to the surface of the tobacco leaf, relative to a total of (i) a weight of the tobacco leaf and (ii) a weight of water used in immersion of the tobacco leaf, water added to the tobacco leaf, or water adhering to the surface of the tobacco leaf. An amount of water contained in a tobacco leaf can be calculated by a known method.

**[0021]** In the heating step, in a case where a tobacco leaf is immersed in water or water is added to a tobacco leaf, a mass ratio of the tobacco leaf to the water (tobacco leaf/water) can be not less than 1/25 and not more than 1/2. This range is preferable because, in a case where the mass ratio is in this range, water can always be in contact with the surface of the tobacco leaf during the heating.

[0022] A heating temperature in the heating step is not lower than 80°C, preferably not lower than 90°C, and more preferably not lower than 95°C. In a case where the tobacco leaf is a fresh leaf, the heating temperature is preferably not higher than 150°C, and more preferably not higher than 120°C. In a case where the tobacco leaf is a cured leaf, the heating temperature is preferably not higher than 150°C, more preferably not higher than 120°C, and even more preferably not higher than 100°C. In a case where the tobacco leaf is a cured leaf, the heating temperature can preferably be 100°C or not higher than 100°C. Also in a case where the tobacco leaf is a fresh leaf, the heating temperature can preferably be 100°C or not higher than 100°C but can also be not lower than 100°C. In a case where the heating temperature is in such a range, it is possible to further increase an amount of solanone in a tobacco leaf. In the present specification, the heating temperature indicates a set temperature of a heating device or the like, but during the heating time, the tobacco leaf itself reaches a temperature hardly different from the set temperature. As such, a "heating temperature" in the present specification means a temperature at which the tobacco leaf is heated.

[0023] In a case where a tobacco leaf is heated at a

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temperature of not lower than 80°C and not higher than 100°C, it is preferable that the tobacco leaf be heated under atmospheric pressure, from the viewpoint of achieving safety and cost reduction without the need of a special device. It is also preferable to heat the tobacco leaf under a condition where oxygen is always present. "Under a condition where oxygen is always present" refers to a state in the heating step in which oxygen is in contact with the tobacco leaf or with water which is in contact with the tobacco leaf. For example, the tobacco leaf can be heated in an open system in an air atmosphere, or can be heated in a closed system while oxygen is added thereto. Note that in the present specification, "in an air atmosphere" means that the heating is carried out in an open system.

**[0024]** To heat the tobacco leaf at a temperature higher than 100°C, a known method can be used to achieve a temperature higher than 100°C. Examples of a method of achieving a temperature higher than 100°C include a method in which heating is carried out under pressure in a closed system.

**[0025]** A heating time in the heating step is not less than 30 minutes and preferably not less than 40 minutes. In a case where the heating time is in such a range, it is possible to further increase an amount of solanone in a tobacco leaf. Further, from the viewpoint of maintaining a high solanone content, the heating time is preferably not more than 120 minutes and more preferably not more than 100 minutes.

[0026] In the increasing method in accordance with an aspect of the present invention, a method of heating is not particularly limited provided that a desired heating temperature is achieved in a state where sufficient water can be present on the surface of the tobacco leaf. Examples of the method of heating include a method in which a tobacco leaf put in a suitable container is heated with use of a hot water bath, a curing oven, a steamer, an incubator, or the like. In a case where a tobacco leaf and water are added to a container and heated, it is preferable to put a lid lightly on the container when heating, in order to prevent water from evaporating out of the container. In a case where (i) a tobacco leaf is immersed in water or water is added to a tobacco leaf and (ii) the tobacco leaf is put in a space filled with steam and is heated, water may be added to and evaporated from, for example, a container that is different from the container in which the tobacco leaf and the water are put.

**[0027]** In the increasing method in accordance with an aspect of the present invention, a tobacco leaf may be stirred with use of, for example, a blade and vibration in order for the surface of the tobacco leaf to be efficiently in contact with water and oxygen.

#### <Another step>

**[0028]** The increasing method in accordance with an aspect of the present invention can include another step either before or after the heating step. The another step is

not particularly limited provided that an effect of the present invention is not compromised. Examples of the step carried out before the heating step include an adjustment step of adjusting the tobacco leaf to be in a desired state and form. Examples of the step carried out after the heating step include a curing step of subjecting, to a curing treatment, the tobacco leaf which has been subjected to the heating step.

**[0029]** As described above, a tobacco leaf that has been subjected to the above-described increasing method is a tobacco leaf having an increased amount of solanone. As such, a tobacco product produced by using, as a material, a tobacco leaf that has been subjected to the above-described increasing method provides tobacco smoke having an improved mouth-feel. Therefore, a tobacco leaf that has been subjected to the above-described increasing method is suitably used as a material for a tobacco product.

[2. Method for producing tobacco material]

**[0030]** A method for producing a tobacco material in accordance with an aspect of the present invention (hereinafter, simply referred to as "production method") includes: a heating step of heating a tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water; and a curing step of subjecting, to a curing treatment, the tobacco leaf which has been subjected to the heating step. This configuration increases an amount of solanone in the tobacco leaf and makes it possible to produce a tobacco material having a high solanone content.

**[0031]** In the present specification, "tobacco material" means a tobacco leaf in the meaning of a material used in production of a tobacco product. The form of a tobacco material can be the same as the above-described form of a tobacco leaf.

**[0032]** Further, the production method in accordance with an aspect of the present invention makes it possible to also increase other aroma and taste components in the tobacco leaf. Examples of such aroma and taste components include β-damascenone, 3-oxo-α-ionol, and blumenol C. Thus, the production method in accordance with an aspect of the present invention makes it possible to produce a tobacco material that is increased not only in amount of solanone but also in amounts of other aroma and taste components and thus is rich in various aroma and taste components.

50 <Heating step>

**[0033]** The heating step is as described above on the heating step in [1. Method for increasing amount of solanone in tobacco leaf].

<Curing step>

[0034] The curing step is a step of subjecting, to a

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curing treatment, a tobacco leaf which has been subjected to the heating step. Since solanone is present on the surface of the leaf, it is expected that solanone in the tobacco leaf is absorbed, though in a very small amount, by water adhering to the surface of the tobacco leaf. As such, leaving the water adhering to the surface of the tobacco leaf risks a decrease in amount of solanone in the tobacco leaf. By including the curing step, it is possible to reduce an escape of solanone from the tobacco leaf in which the amount of solanone has been increased, and to thereby produce a tobacco material maintaining a high solanone content. The curing treatment can be carried out by a known method in accordance with a state and form of the tobacco leaf subjected to the heating step. Examples of a curing method carried out in a case where a fresh leaf is subjected to the heating step in the form of a tobacco leaf itself include, but are not limited to, air curing, fire curing, flue curing, and sun curing. The air curing involves, but is not limited to, hanging a tobacco leaf in a well-ventilated barn and airing the tobacco leaf for 4 to 8 weeks to cure the tobacco leaf. The fire curing involves hanging a tobacco leaf in a large barn and heatcuring the tobacco leaf by fire continuously or intermittently for 3 days to 10 weeks depending on the step and the tobacco leaf. The flue curing involves hanging tobacco leaves arranged in a row in a curing shed and gradually increasing the temperature, typically over a span of approximately 1 week, to cure the tobacco leaves. The sun curing involves curing a tobacco leaf under the sun.

#### <Another step>

[0035] The production method in accordance with an aspect of the present invention can include another step either (i) before the heating step, (ii) between the heating step and the curing step, or (iii) after the curing step. The another step is not particularly limited provided that an effect of the present invention is not compromised. Examples of the step carried out before the heating step include an adjustment step of adjusting the tobacco leaf to be in a desired state and form. Examples of the step carried out after the curing step include a processing step of processing the tobacco leaf into a form of a desired tobacco material. As such, in the production method in accordance with an aspect of the present invention, a tobacco leaf that has been subjected to the curing step can be used as a tobacco material, or a tobacco leaf that has been further subjected to a processing treatment can be used as a tobacco material.

**[0036]** Thus, the production method described above makes it possible to obtain a tobacco material having a high solanone content. The tobacco material obtained by the production method in accordance with an aspect of the present invention can be used in production of any tobacco product. For example, the tobacco material can be used for producing (i) a smoking product such as heatnot-burn tobacco, a paper-wrapped cigarette, a cigar, and pipe smoking tobacco and (ii) a smokeless tobacco

product such as snuff tobacco and chewing tobacco.

#### [3. Method for extracting solanone]

[0037] A method for extracting solanone in accordance with an aspect of the present invention (hereinafter, simply referred to as "extraction method") includes: a heating step of heating a tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water; and an extraction step of extracting solanone from the tobacco leaf which has been subjected to the heating step. According to this configuration, extraction is carried out from a tobacco leaf in which an amount of solanone has been increased. This makes it possible to extract a greater amount of solanone in comparison to solanone obtained by a conventional method.

### <Heating step>

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**[0038]** The heating step is as described above on the heating step in [1. Method for increasing amount of solanone in tobacco leaf].

#### <Extraction step>

[0039] The extraction step is a step of extracting solanone from a tobacco leaf which has been subjected to the heating step. Extraction of solanone can be carried out by a known method. For example, a tobacco leaf which has been subjected to the heating step is put in a suitable container, an organic solvent such as hexane or water is further added, and the mixture is shaken. This makes it possible to extract solanone from the tobacco leaf which has been subjected to the heating step. Further, it is also possible to use a supercritical carbon dioxide extraction method or the like. The organic solvent used is not limited to a particular one provided that the organic solvent is a solvent that makes it possible to extract solanone from a tobacco leaf. Examples of the organic solvent used include hexane, ether, ethanol, ethyl acetate, acetone, glycerin, and propylene glycol. An amount of the organic solvent added can be set as appropriate by a person skilled in the art, and can be, for example, 5 times to 50 times the amount of the tobacco leaf in mass ratio, and preferably 20 times to 30 times the amount of the tobacco leaf in mass ratio.

### <Another step>

**[0040]** The extraction method in accordance with an aspect of the present invention can include another step either (i) before the heating step, (ii) between the heating step and the extraction step, or (iii) after the extraction step. The another step is not particularly limited provided that an effect of the present invention is not compromised. Examples of the step carried out before the heating step include an adjustment step of adjusting the

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tobacco leaf to be in a desired state and form. Examples of the step carried out after the extraction step include a step of removing an unwanted component, a step of concentrating an extract, and a step of drying an extract. [0041] The solanone obtained by the extraction method in accordance with an aspect of the present invention can be used in production of any tobacco product. Further, the solanone can also be used in applications in which solanone is required, other than tobacco products.

**[0042]** Aspects of the present invention can also be expressed as follows:

A method for increasing an amount of solanone in a tobacco leaf in accordance with Aspect 1 of the present invention includes: a heating step of heating a tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water.

[0043] A method for increasing an amount of solanone in a tobacco leaf in accordance with Aspect 2 of the present invention is preferably configured such that, in Aspect 1 described above, the tobacco leaf is a fresh leaf. [0044] A method for increasing an amount of solanone in a tobacco leaf in accordance with Aspect 3 of the present invention is preferably configured such that, in Aspect 1 described above, the tobacco leaf is a cured leaf.

**[0045]** A method for increasing an amount of solanone in a tobacco leaf in accordance with Aspect 4 of the present invention is preferably configured such that, in any one of Aspects 1 to 3 described above, the heating step includes heating the tobacco leaf at a temperature of not lower than 80°C and not higher than 100°C under atmospheric pressure.

**[0046]** A method for producing a tobacco material in accordance with Aspect 5 of the present invention is preferably configured such that the method includes: a heating step of heating a tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water; and a curing step of subjecting, to a curing treatment, the tobacco leaf which has been subjected to the heating step.

**[0047]** A method for extracting solanone in accordance with Aspect 6 of the present invention is preferably configured such that the method includes: a heating step of heating a tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water; and an extraction step of extracting solanone from the tobacco leaf which has been subjected to the heating step.

**[0048]** The present invention is not limited to the embodiments, but can be altered by a skilled person in the art within the scope of the claims. The present invention also encompasses, in its technical scope, any embodiment derived by combining technical means disclosed in differing embodiments.

Examples

**[0049]** The following description will discuss Examples of the present invention. Note that all of the tobacco plants indicated in Examples below are *Nicotiana tabacum*.

[Example 1: consideration of treatment time]

[0050] 0.2 g of powder of a cured leaf of tobacco (variety: Mizufu) ground with a mill was put in each of 50-mL centrifugation tubes, had 5 mL of water added thereto, and was heated to 100°C under atmospheric pressure with use of a curing oven (AFO-80 (manufactured by IWAKI GLASS CO., LTD.). In order to prevent evaporation of water from the centrifugation tube during heating with use of the curing oven, a lid of aluminum foil was lightly put on the centrifugation tube. Sampling was carried out at predetermined intervals from a start of the heating until 120 minutes after the start of the heating. Every time the predetermined interval has passed, the centrifugation tubes were taken out and cooled, one centrifugation tube at a time, 5 mL of hexane was added, and the mixture was shaken for 30 minutes. After the shaking, the mixture was left to stand still for 5 minutes, and a hexane layer in an upper layer was analyzed with a gas chromatography mass spectrometer (GC/MS, Shimadzu QP2010 Ultra, manufactured by Shimadzu Corporation). In parallel, a test section to be heated without addition of water was set.

**[0051]** Results are shown in Fig. 1. In Fig. 1, a vertical axis indicates a relative value of an amount of solanone in a case where an amount of solanone as of the start of the treatment is assumed to be 100, and a horizontal axis indicates a treatment time (min). In the test section to which water had been added, the amount of solanone started to increase 20 minutes after the start of the heating, was maximized 50 minutes to 60 minutes after the start of the heating, and then the high solanone content was maintained until 120 minutes after the start of the heating. In the test section heated without addition of water, the amount of solanone hardly increased.

[Example 2: consideration of material]

[0052] A fresh leaf of each of seven tobacco varieties indicated below was cut into an approximately 5-cm square piece, and the piece was further shredded into a size that fits in a centrifugation tube. The shreds of the fresh leaf of each variety were put in an amount of 1 g into a 50-mL centrifugation tube, 10 mL of water was added thereto, and the shreds of the fresh leaf were subjected to a hot water bath for 60 minutes in boiling water under atmospheric pressure. Then, each centrifugation tube was taken out and cooled, 5 mL of hexane was added thereto, and the mixture was shaken for 30 minutes. After the shaking, the mixture was left to stand still for 5 minutes, and a hexane layer in an upper layer was

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analyzed with GC/MS for comparison with an amount of solanone in a sample that had not been subjected to a heat treatment.

(Tobacco varieties)

#### [0053]

Comstock Spanish
Okinawa
Amarelinho Dechicoana
Perevi
Cuba Prieto
Basma
Aromatique 272

**[0054]** Results are shown in Fig. 2. In Fig. 2, a vertical axis indicates a relative value of an amount of solanone in a case where an amount of solanone in a sample obtained by heating Basma is assumed to be 1, and a horizontal axis indicates each variety. The amount of solanone increased in all of the seven tobacco varieties in comparison with the sample not subjected to a heat treatment.

[Example 3: consideration of treatment temperature]

**[0055]** 0.2 g of powder of a cured leaf of tobacco (variety: Mizufu) ground with a mill was put in each of 50-mL centrifugation tubes, had 5 mL of water added thereto, and was heated at room temperature (20°C), 60°C, 80°C, or 100°C for 60 minutes under atmospheric pressure. For the test section heated at 60°C, a thermohygrostat (ESPEC CORP. (PR-4J)) was used. For the test sections respectively heated at 80°C and 100°C, a curing oven was used similarly as in Example 1. Then, each centrifugation tube was taken out and cooled, 5 mL of hexane was added thereto, and the mixture was shaken for 30 minutes. After the shaking, the mixture was left to stand still for 5 minutes, and a hexane layer in an upper layer was analyzed with GC/MS.

**[0056]** Results are shown in Fig. 3. In Fig. 3, a vertical axis indicates a relative value of an amount of solanone in a case where an amount of solanone in an untreated sample which has not been subjected to a heat treatment is assumed to be 1, and a horizontal axis indicates a treatment temperature (°C). The test section heated at 80°C had an increased amount of solanone in comparison with the sample not subjected to a heat treatment, and the test section heated at 100°C had an increased amount of solanone by not less than 9 times in comparison with the sample not subjected to a heat treatment. In the test sections heated respectively at 20°C and 60°C, the amount of solanone hardly increased in comparison with the sample not subjected to a heat treatment.

[Example 4: consideration of treatment method]

[0057] 0.2 g of powder of a cured leaf of tobacco (variety: Mizufu) ground with a mill was put in each of 50-mL centrifugation tubes, had 0.5 mL of water, 5 mL of water, or no water further added thereto, and was heated at 100°C for 60 minutes under atmospheric pressure. Each test section was heated with use of a curing oven similarly as in Example 1. Further, also set was another test section to which 0.5 mL of water was added and which was heated with use of a steamer. In the case of heating with use of a steamer, the heating was carried out by a method described below. Specifically, a centrifugation tube containing a sample and predetermined water was put in a steamer, and water was introduced into the steamer up to approximately 5 cm from the bottom of the steamer. A lid was placed on the steamer, and the water in the steamer was boiled by heating with an IH heater. The centrifugation tube was placed upright in the steamer, and aluminum foil was used as the lid lightly put on the centrifugation tube. Then, each centrifugation tube was taken out and cooled, 5 mL of hexane was added thereto, and the mixture was shaken for 30 minutes. After the shaking, the mixture was left to stand still for 5 minutes, and a hexane layer in an upper layer was analyzed with GC/MS. Further, an untreated section not subjected to a heat treatment was also set.

**[0058]** Results are shown in Fig. 4. In Fig. 4, a vertical axis indicates a relative value of an amount of solanone in a case where an amount of solanone in the untreated section not subjected to a heat treatment is assumed to be 1, and a horizontal axis indicates each method of heating. Regardless of the method of heating, each of the test sections to which water had been added had an increased amount of solanone, by 3 times to 7 times in comparison with the untreated section. The test section having the greatest increase in amount of solanone was the test section that had 0.5 mL of water added and was heated with use of an oven.

[Example 5: consideration of change in other components]

[0059] 0.2 g of powder of a cured leaf of tobacco (variety: Mizufu) ground with a mill was put in each of 50-mL centrifugation tubes, had 0.5 mL of water or no water further added thereto, and was heated at 100°C for 60 minutes under atmospheric pressure. The heating was carried out with use of a curing oven similarly as in Example 1. Then, each centrifugation tube was taken out and cooled, 5 mL of hexane was added thereto, and the mixture was shaken for 30 minutes. After the shaking, the mixture was left to stand still for 5 minutes, and a hexane layer in an upper layer was analyzed with GC/MS. Further, an untreated section not subjected to a heat treatment was also set.

**[0060]** Fig. 5 shows results of analysis of  $\beta$ -damascenone and 3-oxo- $\alpha$ -ionol. In Fig. 5, a vertical axis indicates

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a relative value of an amount of  $\beta$ -damascenone or an amount of  $3\text{-}oxo\text{-}\alpha\text{-}\mathrm{ionol}$  in a case where an amount of  $\beta$ -damascenone and an amount of  $3\text{-}oxo\text{-}\alpha\text{-}\mathrm{ionol}$  in the untreated section not subjected to a heat treatment were each assumed to be 1, and a horizontal axis indicates each treatment method. The test section to which water had been added had increased amounts of  $\beta$ -damascenone and  $3\text{-}oxo\text{-}\alpha\text{-}\mathrm{ionol}$  in comparison with the untreated section. By contrast, the test section which had been heated without addition of water exhibited no increase in amounts of  $\beta$ -damascenone and  $3\text{-}oxo\text{-}\alpha\text{-}\mathrm{ionol}$ , in comparison with the untreated section.

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Industrial Applicability

**[0061]** The present invention is applicable to production of a tobacco material.

Claims 20

- 1. A method for increasing an amount of solanone in a tobacco leaf, the method comprising: a heating step of heating a tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water.
- **2.** The method as set forth in claim 1, wherein the tobacco leaf is a fresh leaf.
- **3.** The method as set forth in claim 1, wherein the tobacco leaf is a cured leaf.
- 4. The method as set forth in any one of claims 1 to 3, wherein the heating step includes heating the tobacco leaf at a temperature of not lower than 80°C and not higher than 100°C under atmospheric pressure.
- 5. A method for producing a tobacco material, the method comprising: a heating step of heating a 40 tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water; and a curing step of subjecting, to a curing treatment, the tobacco leaf which has been subjected to the heating step.
- **6.** A method for extracting solanone, the method comprising: a heating step of heating a tobacco leaf for not less than 30 minutes at a temperature of not lower than 80°C in the presence of water; and an extraction step of extracting solanone from the tobacco leaf which has been subjected to the heating step.

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FIG. 1

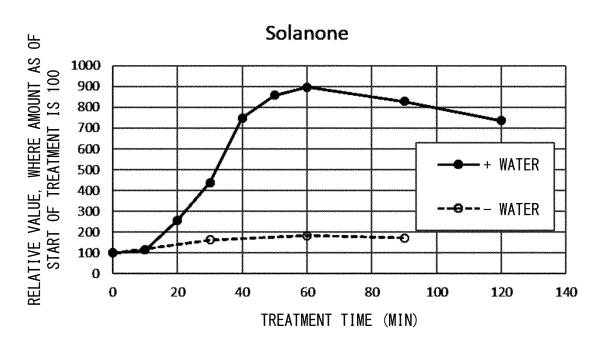
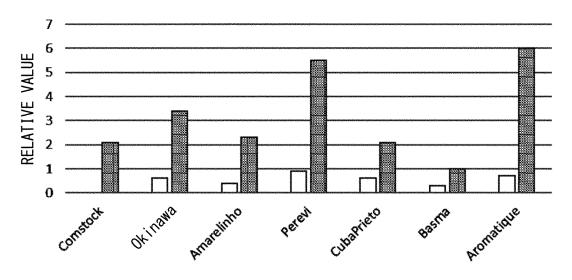


FIG. 2

# Solanone



□- TREATMENT 🖼+ TREATMENT

FIG. 3
Solanone

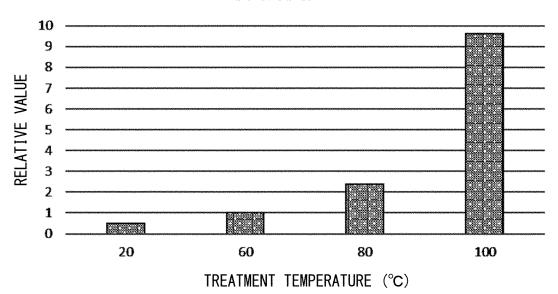
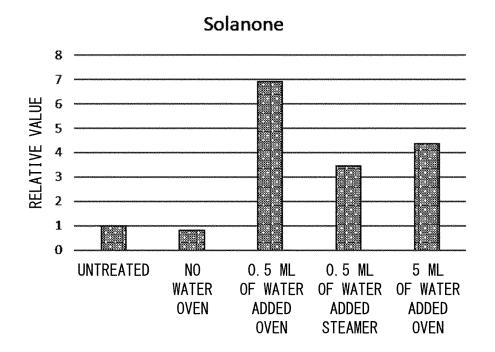
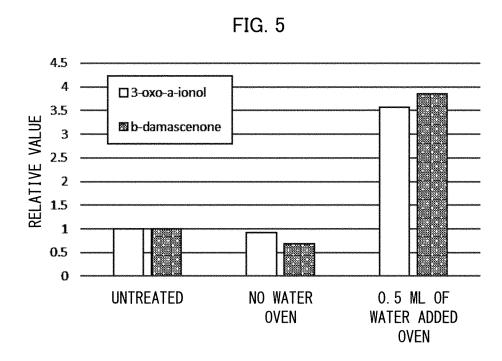


FIG. 4





#### INTERNATIONAL SEARCH REPORT International application No. PCT/JP2023/005224 5 CLASSIFICATION OF SUBJECT MATTER **A24B 15/24**(2006.01)i; **A24B 3/12**(2006.01)i FI: A24B3/12 Z; A24B15/24 According to International Patent Classification (IPC) or to both national classification and IPC 10 FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) A24B15/24; A24B3/12 Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched 15 Published examined utility model applications of Japan 1922-1996 Published unexamined utility model applications of Japan 1971-2023 Registered utility model specifications of Japan 1996-2023 Published registered utility model applications of Japan 1994-2023 Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) 20 DOCUMENTS CONSIDERED TO BE RELEVANT Category\* Citation of document, with indication, where appropriate, of the relevant passages Relevant to claim No. 1\_5 EP 3895550 A1 (KT & G CORPORATION) 20 October 2021 (2021-10-20) X 25 paragraphs [0043]-[0063], tables 1, 2 Y 6 JP 53-133700 A (JAPAN TOBACCO & SALT PUBLIC CORP.) 21 November 1978 X 5 (1978-11-21) column 7, line 8 to column 8, line 8 30 Y JP 2020-517277 A (BRITISH AMERICAN TOBACCO (INVESTMENTS) LTD.) 18 June 6 2020 (2020-06-18) paragraph [0051] WO 2014/060956 A1 (SOUZA CRUZ S.A.) 24 April 2014 (2014-04-24) 1-6 Α Α JP 10-66559 A (R J REYNOLDS TOBACCO CO.) 10 March 1998 (1998-03-10) 1-6 35 JP 9-224630 A (JAPAN TOBACCO INC.) 02 September 1997 (1997-09-02) 1-6 A A JP 2018-7687 A (R J REYNOLDS TOBACCO CO.) 18 January 2018 (2018-01-18) 1-6 40 See patent family annex. Further documents are listed in the continuation of Box C. Special categories of cited documents: 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 document defining the general state of the art which is not considered to be of particular relevance document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone earlier application or patent but published on or after the international filing date document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) 45 document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art document referring to an oral disclosure, use, exhibition or other document published prior to the international filing date but later than the priority date claimed "&" document member of the same patent family Date of the actual completion of the international search Date of mailing of the international search report 50 30 March 2023 11 April 2023 Name and mailing address of the ISA/JP Authorized officer Japan Patent Office (ISA/IP) 3-4-3 Kasumigaseki, Chiyoda-ku, Tokyo 100-8915 55

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