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(54) **ORAL TOBACCO PRODUCT, METHOD FOR MANUFACTURING PACKAGING MATERIAL FOR ORAL TOBACCO PRODUCT, AND METHOD FOR MANUFACTURING ORAL TOBACCO PRODUCT**

(57) Provided is an oral tobacco product that includes, as constituent elements, a tobacco filler and a liquid-permeable packaging material for packaging the tobacco filler, wherein the packaging material includes a tobacco material, and the weight of solids in the packaging material is 11.0 g/m<sup>2</sup> or greater.

**EP 3 777 568 A1**

## Description

[Technical Field]

5     **[0001]** The present invention relates to an oral tobacco product, a method for manufacturing a packaging material for an oral tobacco product and a method for manufacturing an oral tobacco product.

[Background Art]

10    **[0002]** Snus, which is one of oral tobacco products, is a package in which a tobacco filler is accommodated in a packaging material formed from a material such as a non-woven fabric, and a user uses it by putting it in his or her oral cavity.

**[0003]** When an oral tobacco product is put into the user's oral cavity, a flavor component derived from a tobacco filler exudes outside of the packaging material and thus the flavor component is delivered to the user.

15    **[0004]** Regarding the packaging material for packaging a tobacco filler, an insoluble fibrous flavored wrapper is known (Patent Document 1). It is described that this fibrous flavored wrapper includes an internal filling material containing non-tobacco plant material fibers.

**[0005]** On the other hand, Patent Document 2 describes an oral tobacco product in which a wet tobacco material is packaged with a packaging material colored with a coloring agent. Brown is exemplified as the color of the coloring agent.

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[Prior Art Document]

[Patent Document]

25     **[0006]**

      [Patent Document 1] Japanese Translation of PCT Application No. 2010-521957

      [Patent Document 2] European Patent No. 2976951

30     [Summary of Invention]

[Technical Problem]

35     **[0007]** A technology in which an oral tobacco product is produced using a packaging material containing fibers derived from a plant different from tobacco, and thus the flavor and taste when using the oral tobacco product are able to be adjusted and a technology in which an oral tobacco product is produced using a packaging material to which a coloring agent is added to exhibit a brownish-colored appearance and thus the appearance of the oral tobacco product is made to have the same color as a general snus product are known.

40     **[0008]** However, a technology for quickly delivering the characteristic flavor and taste of a tobacco material to a user with a focus on a packaging material has not been disclosed.

**[0009]** In view of the above circumstances, an object of the present invention is to provide an oral tobacco product that can quickly deliver the characteristic flavor and taste of a tobacco material to a user when used.

[Solution to Problem]

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**[0010]** The inventors conducted extensive studies and as a result, found that it is possible to achieve the above object using a packaging material for packaging a tobacco filler, which contains a tobacco material so that the weight of solids is 11.0 g/m<sup>2</sup> or greater.

**[0011]** Specifically, the present invention is as follows.

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      [1] An oral tobacco product including, as constituent elements, a tobacco filler and a liquid-permeable packaging material for packaging the tobacco filler, wherein the packaging material includes solids derived from a tobacco material and a weight of the solids in the packaging material is 11.0 g/m<sup>2</sup> or greater.

55     [2] The oral tobacco product according to [1], wherein the liquid-permeable packaging material includes a green part having an a\* value of 1.0 or less in a CIELab color system.

      [3] The oral tobacco product according to [1] or [2], wherein the liquid-permeable packaging material is composed of a non-woven fabric.

      [4] The oral tobacco product according to any of [1] to [3], wherein the packaging material is obtained by adding

thereto any of the following 1) to 3) as a tobacco material:

- 1) a tobacco powder extraction liquid obtained using a solvent containing ethanol and/or propylene glycol as an extraction solvent;
- 2) a slurry mixture containing a tobacco powder having an average particle size of 30  $\mu\text{m}$  or less and a liquid dispersion medium; and
- 3) a tobacco powder.

[5] The oral tobacco product according to [4], wherein an  $a^*$  value of the tobacco powder is 1.0 or less, and an activity value of an enzyme responsible for browning of the tobacco powder is 0.1 U/g or less.

[6] The oral tobacco product according to any of [1] to [5], wherein the tobacco filler has a green color having an  $a^*$  value of 1.0 or less in a CIELab color system.

[7] A method for manufacturing a packaging material for an oral tobacco product, the method including a step of adding a tobacco material to a liquid-permeable packaging material, wherein the step is a step of adding the tobacco material to the liquid-permeable packaging material so that a weight of solids of the tobacco material in the packaging material is 11.0 g/m<sup>2</sup> or greater.

[8] The method for manufacturing a packaging material for an oral tobacco product according to [7], wherein the liquid-permeable packaging material is a non-woven fabric.

[9] The method for manufacturing a packaging material for an oral tobacco product according to [7] or [8], wherein the tobacco material is a slurry mixture containing a tobacco powder having an average particle size of 30  $\mu\text{m}$  or less and a liquid dispersion medium.

[10] The method for manufacturing a packaging material for an oral tobacco product according to [7] or [8], wherein an extraction liquid obtained by a step of extracting a tobacco powder using a solvent containing ethanol and/or propylene glycol as an extraction solvent is used as the tobacco material.

[11] The method for manufacturing a packaging material for an oral tobacco product according to [10], wherein a weight ratio between the tobacco powder and the solvent in the extraction liquid is 1:1.5 to 1:8.

[12] The method for manufacturing a packaging material for an oral tobacco product according to any of [9] to [11], wherein the tobacco powder is obtained through an alkaline sterilization treatment.

[13] The method for manufacturing a packaging material for an oral tobacco product according to any of [9] to [12], wherein the step of adding the tobacco material to the liquid-permeable packaging material is performed by immersion, spraying, or printing.

[14] The method for manufacturing a packaging material for an oral tobacco product according to [8], wherein the addition of the tobacco material is performed by adding a tobacco powder during papermaking of the non-woven fabric.

[15] A method for manufacturing an oral tobacco product, the method including a step of filling the packaging material obtained by the method for manufacturing a packaging material for an oral tobacco product according to any of [7] to [14] with a tobacco filler.

[16] The method for manufacturing an oral tobacco product according to [15], wherein the following steps are included, as a step of manufacturing the tobacco filler, before the step of filling the packaging material with the tobacco filler:

- 1) a step of mixing a tobacco powder, a salt, and a base;
- 2) a step of heating the mixture obtained through the step 1);
- 3) a step of cooling and drying the mixture after heating; and
- 4) a step of adding an additive to the mixture after drying.

[17] The method for manufacturing an oral tobacco product according to [16], wherein when the tobacco material added to the packaging material is an extraction liquid obtained by a step of extracting a tobacco powder using a solvent containing ethanol and/or propylene glycol as an extraction solvent, the method includes a step of adding an extraction residue obtained in the extraction step to the tobacco filler.

[18] The method for manufacturing an oral tobacco product according to [16] or [17], wherein the following steps a) and b) are included, as a step of manufacturing the tobacco powder used for manufacturing the tobacco filler, before manufacturing the tobacco filler, and a water content of the tobacco powder is kept at 7 weight% or less after the step a) until the step b) and the step 1):

- a) a step of drying tobacco leaves after harvesting the tobacco leaves until a water content of the tobacco leaves is 7 weight% or less; and
- b) a step of obtaining a tobacco powder by crushing the tobacco leaves after the step of drying.

[19] A method for manufacturing an oral tobacco product, the method including: a step of filling a liquid-permeable packaging material with a tobacco filler having a green color having an a\* value of 1.0 or less in a CIELab color system; and a step of adding a solvent containing ethanol and/or propylene glycol to a package after the filling from outside of the package.

[20] The method for manufacturing an oral tobacco product according to [19], wherein the following steps are included, as a step of manufacturing the tobacco filler, before the step of filling the packaging material with the tobacco filler:

- 1) a step of mixing a tobacco powder, a salt, and a base;
- 2) a step of heating the mixture obtained through the step 1);
- 3) a step of cooling and drying the mixture after heating; and
- 4) a step of adding an additive to the mixture after drying.

[21] The method for manufacturing an oral tobacco product according to [20], wherein the following steps a) and b) are included, as a step of manufacturing the tobacco powder used for manufacturing the tobacco filler, before manufacturing the tobacco filler, and a water content of the tobacco powder is kept at 7 weight% or less after the step a) until the step b) and the step 1):

- a) a step of drying tobacco leaves after harvesting the tobacco leaves until a water content of the tobacco leaves is 7 weight% or less; and
- b) a step of obtaining a tobacco powder by crushing the tobacco leaves after the step of drying.

#### [Effects of Invention]

**[0012]** According to the present invention, it is possible to provide an oral tobacco product and the like in which the characteristic flavor and taste of a tobacco material are quickly delivered to a user after the user puts the product in his or her oral cavity.

#### [Brief Description of Drawings]

#### **[0013]**

[Fig. 1]

Fig. 1 is a flowchart showing an example of a method for manufacturing a packaging material containing a tobacco material.

[Fig. 2]

Fig. 2 is a flowchart showing an example of a method for manufacturing a tobacco product.

[Fig. 3]

Fig. 3 is a flowchart showing an example of a method for preparing a tobacco powder.

[Fig. 4]

Fig. 4 is a flowchart showing an example of a method for preparing a tobacco filler.

#### [Description of Embodiments]

**[0014]** Hereinafter, the present invention will be described in detail with reference to embodiments, examples and the like, but the present invention is not limited to the following embodiments, examples, and the like, and can be arbitrarily changed and implemented without departing from the spirit and scope of the present invention.

#### <Tobacco product>

**[0015]** A tobacco product according to an embodiment of the present invention is an oral tobacco product including, as constituent elements, a tobacco filler and a liquid-permeable packaging material for packaging a tobacco filler (hereinafter simply referred to as a packaging material), wherein a tobacco material is added to the packaging material, and in the packaging material, the weight of solids is 11.0 g/m<sup>2</sup> or greater.

**[0016]** The weight of the solids in the packaging material is defined as follows.

[Weight of solids (g/m<sup>2</sup>) = [weight of packaging material including solids (g/m<sup>2</sup>)] - [weight of packaging material after solids are removed (g/m<sup>2</sup>)]

5 [Weight of packaging material including solids (g/m<sup>2</sup>)]

[0017] After a tobacco filler is separated from a packaging material of a sample, the tobacco filler adhered to the inner surface of the packaging material is cleaned off with an air gun (a pressure of 0.6 MPa and a nozzle inner diameter of 3 mm) for about 10 seconds, it is visually confirmed that no tobacco filler is adhered to the inner surface of a non-woven fabric, and the material is then cut into 2 cm×2 cm pieces and dried under conditions of 40°C for 2 hours, and the weight is then measured. The weight of 10 pieces of 2 cm×2 cm cut product is measured.

[0018] The total weight of 10 pieces is divided by the total area of 10 pieces to obtain the weight of the packaging material including solids (g/m<sup>2</sup>).

15 [Weight of packaging material after solids are removed (g/m<sup>2</sup>)]

[0019] 50 ml of distilled water is added to 10 pieces of a 2 cm×2 cm packaging material of which the weight has been measured above, ultrasonic washing is performed at room temperature (22°C) for 10 minutes and drying is then performed under conditions of 40°C and for 12 hours, and the weight is then measured.

20 [0020] The total weight of 10 pieces is divided by the total area of 10 pieces to obtain the weight of the packaging material after the solids are removed (g/m<sup>2</sup>). Here, when the size of the packaging material is less than 2 cm×2 cm, the size can be appropriately adjusted and used as a measurement sample.

Specific examples of oral tobacco products include snus.

25 [0021] The tobacco filler includes a tobacco powder and water.

[0022] The tobacco filler may further include a filling material in order to adjust flavor and taste of the oral tobacco product and/or adjust color of the tobacco filler.

[0023] In addition, when the tobacco filler is obtained through a subsequent alkaline sterilization treatment, it can further include a salt and a base.

[0024] The tobacco powder can include shredded lamina of dried tobacco leaves, fine powder, fibers and the like, and can be prepared by the method to be described below. In this specification, tobacco leaves may include mesophyll (lamina), veins (stems), and roots. The above tobacco filler can include components derived from stems of tobacco leaves and roots in addition to tobacco powder obtained substantially from the lamina of tobacco leaves.

35 [0025] The particle size of the tobacco powder is not particularly limited, but in order to enhance familiarity in the oral cavity, improve a feeling of use, and improve release of a flavor component contained in the tobacco powder into the oral cavity, the size is preferably one that allows passage through a 1.2 mm mesh and more preferably one that allows passage through a 1.0 mm mesh.

[0026] Tobacco species used as a raw material of the tobacco powder are not particularly limited, and examples thereof include species of the genus *Nicotiana*, such as *Nicotiana tabacum* yellow species, Burley species, and *Nicotiana rustica* Brasilia species. The same species can be used for tobacco materials and tobacco leaves to be described below.

[0027] The tobacco filler of the tobacco product is preferably green with an a\* value of 1.0 or less expressed by a CIELab color system method. In order to realize this, a tobacco powder used in the tobacco filler having a green color having an a\* value of 1.0 or less expressed by the CIELab color system method can be exemplified. This point will be specifically described in the section of the following method for manufacturing a tobacco product.

[0028] Examples of filling materials that can be contained in the oral tobacco product according to the embodiment of the present invention include a cellulose powder. The average particle size of the cellulose powder is, for example, 20 to 500 μm, and is preferably 30 to 100 μm.

[0029] The weight proportion of the (dried) tobacco powder contained in the tobacco filler according to the embodiment of the present invention can be, for example, 35 to 90 weight%, and is preferably 45 to 60 weight%, with respect to a total amount of the dried tobacco filler.

[0030] The tobacco filler according to the embodiment of the present invention can contain a tobacco filler. The weight proportion of the (dried) filling material can be for example, 0.1 to 55 weight%, and is preferably 30 to 45 weight%, with respect to a total amount of the dried tobacco filler.

55 [0031] The tobacco filler according to the embodiment of the present invention can contain a salt, and the weight proportion of the salt can be, for example, 0.1 to 12 weight%, and is preferably 3.0 to 8.0 weight%, with respect to a total amount of the dried tobacco filler. The type of the salt can include one or more selected from the group consisting of sodium chloride, potassium chloride, sodium citrate, potassium citrate, sodium acetate, and potassium acetate. Among

these, at least sodium chloride is preferably used.

**[0032]** The tobacco filler according to the embodiment of the present invention can contain a base, and the weight proportion of the base can be, for example, 0.1 to 12 weight%, and is preferably 5.0 to 9.0 weight%, with respect to the total amount of the dried tobacco filler. Types of the base include one or more selected from the group consisting of potassium carbonate, sodium carbonate, and sodium bicarbonate. Among these, at least sodium carbonate and/or potassium carbonate is preferably used.

**[0033]** The water content of the tobacco filler contained in the oral tobacco product according to the embodiment of the present invention can be, for example, 5 to 50 weight%, and is preferably 10 to 45 weight%.

**[0034]** The pH of the tobacco filler contained in the oral tobacco product is preferably set to, for example, 8.0 to 9.0. When the tobacco filler has a specific green color to be described below, it is suitable to maintain the color at such a pH.

**[0035]** Here, the pH of the tobacco filler is measured as follows.

<Measurement of pH>

**[0036]** 2.0 g of a tobacco filler to be measured (or a mixture containing a tobacco powder to be described below) is weighed out into a screw tube, 20 ml of distilled water is added, and the mixture is shaken at 200 rpm for 10 minutes and is subjected to an extraction treatment. The obtained extraction liquid is left for 5 minutes and the pH is measured at (25°C) under the following conditions to obtain a pH value of the tobacco filler.

<pH measurement conditions>

**[0037]**

Measurement device: LAQUA F-72 FLAT ISFET pH electrode (commercially available from Horiba Ltd.)

Device calibration: 3-point calibration using a phthalic acid pH standard solution (pH 4.01), a neutral phosphate pH standard solution (pH 6.86), and a borate pH standard solution (pH 9.18) (all commercially available from Wako Pure Chemical Industries, Ltd.)

**[0038]** The oral tobacco product according to the embodiment of the present invention includes a liquid-permeable packaging material for packaging the tobacco filler. A package obtained by packaging a tobacco filler with the packaging material is an oral tobacco product according to the embodiment of the present invention. Regarding a mode in which a tobacco filler is packaged with a packaging material, for example, like a known snus product, a mode in which the periphery of the tobacco filler is covered with a packaging material so that the tobacco filler becomes the content of the oral tobacco product can be exemplified.

**[0039]** The shape of the oral tobacco product can be known shape, and a shape with a length of about 20 to about 45 mm, a width of about 10 to about 20 mm, and a thickness of about 3 to about 6 mm can be exemplified.

**[0040]** The ratio of the length to the width can be about 1.2 to 3.0.

**[0041]** The packaging material includes solids derived from the tobacco material.

**[0042]** In this specification, the solids derived from the tobacco material include the tobacco powder itself and/or solids ascribing to a substance which is contained in a tobacco powder and is solvent-soluble, and can include components (a pigment, a flavor component, etc.) contained in tobacco leaves together with the solids.

**[0043]** In the packaging material, the weight of solids is 11.0 g/m<sup>2</sup> or greater. When the packaging material includes solids at such a weight proportion, the characteristic flavor and taste of the tobacco material can be quickly delivered to the user when the users uses the oral tobacco product according to the embodiment of the present invention.

**[0044]** In the packaging material, the weight of solids contained in the packaging material is more preferably 12.0 g/m<sup>2</sup> or greater and still more preferably 15.0 g/m<sup>2</sup> or greater. The upper limit of the weight of solids contained in the packaging material is not particularly limited, and an extent to which solids do not fall off of the packaging material during transportation of the tobacco product may be exemplified. Specifically, in consideration of actual manufacturing suitability and quality suitability, the upper limit value of the weight of solids added to the packaging material can be, for example, 40.0 g/m<sup>2</sup> or less or 35.0 g/m<sup>2</sup> or less.

**[0045]** The packaging material is liquid-permeable. The liquid-permeable packaging material is a packaging material that does not allow permeation of a particulate substance constituting a tobacco filler that is contained and has a permeability to such an extent that, when a solvent is added from outside of the oral tobacco product, the solvent reaches the tobacco filler, and the substance dissolving in a solvent contained in the tobacco filler exudes to the outside.

**[0046]** Here, the solvent here is a solvent containing ethanol and/or propylene glycol.

**[0047]** Examples of liquid-permeable packaging materials include a non-woven fabric containing a natural fiber such as wood pulp (cellulose) or a synthetic fiber and a binder as constituent elements. Regarding such a non-woven fabric, commercially available products can be used.

**[0048]** The basis weight of the non-woven fabric can be about 10 to 50 g/m<sup>2</sup> and is preferably 15 to 30 g/m<sup>2</sup>.

**[0049]** The liquid-permeable packaging material preferably has a green part having an a\* value of 1.0 or less in the CIELab color system. The green part can be present on a part or all of the surface of the packaging material. For example, cases in which 50% or more, 70% or more in another mode, 80% or more in still another mode, and 90% or more in a preferable mode of the surface area of the packaging material has the green part can be exemplified. In these modes, the green appearance gives the user an impression of freshness. When the packaging material is green, the green color is preferably derived from the tobacco material.

<Method for manufacturing packaging material for oral tobacco product>

**[0050]** Regarding a method for incorporating a tobacco material into a liquid-permeable packaging material (a method for manufacturing a packaging material according to an embodiment of the present invention), for example, three modes shown in Fig. 1 can be exemplified.

**[0051]** Any mode of the method includes a step of adding a tobacco material to a liquid-permeable packaging material, and the step is a step in which a tobacco material is added to a liquid-permeable packaging material so that the weight of solids in the packaging material is 11.0 g/m<sup>2</sup> or greater, preferably 12.0 g/m<sup>2</sup> or greater, and still more preferably 15.0 g/m<sup>2</sup> or greater. The upper limit of the weight of solids is not particularly limited, and can be an extent that solids do not fall off of the packaging material during transportation of the tobacco product. Specifically, the upper limit value of the weight of solids added to the packaging material can be, for example, 40.0 g/m<sup>2</sup> or less or 35.0 g/m<sup>2</sup> or less.

**[0052]** The first mode is a method in which a tobacco powder extraction liquid is prepared, and the extraction liquid is added as a tobacco material to the above packaging material ((1) in Fig. 1). Regarding the packaging material, a non-woven fabric can be exemplified.

**[0053]** In this mode, the tobacco powder can be extracted using a solvent containing ethanol and/or propylene glycol as the solvent. Regarding the solvent, preferably, ethanol or propylene glycol is used alone. The purity is preferably close to 100%, but a very small amount of water that does not interfere with extraction can be mixed in.

**[0054]** The weight ratio between tobacco powder: solvent in this case can be, for example, 1:1.5 to 1:8, and is preferably about 1:1.5 to 1:7. The extraction temperature is, for example, 10°C to 90°C, and preferably 20°C to 85°C. The extraction time is, for example, 10 to 180 minutes, and preferably 30 to 120 minutes.

**[0055]** According to the above extraction, it is possible to obtain a tobacco powder having a fine particle size and an extraction liquid including solvent-soluble solids contained in the tobacco powder.

**[0056]** The extraction residue is separated from the extraction liquid obtained above using an appropriate method such as using a mesh and then added to the above packaging material by an appropriate method. Examples of such methods include a method of immersing a packaging material in an extraction liquid, a method of filling an extraction liquid into a sprayer such as a spraying sprayer and spraying it on a packaging material, and a method of soaking an extraction liquid in a printing instrument such as a sponge roll, pressing the printing instrument against the surface of the packaging material and performing printing.

**[0057]** According to at least one of these methods, the extraction liquid obtained above is added so that the weight of solids in the packaging material is 11.0 g/m<sup>2</sup> or greater.

**[0058]** In this mode, after the above extraction liquid is added, the packaging material is dried, and thus the solvent contained in the extraction liquid is removed. Thereby, solids are deposited inside and on the surface of the packaging material.

**[0059]** Preferably, the tobacco powder to be extracted in this mode is obtained as follows. First, a base is added to and mixed with the tobacco powder obtained in <Preparation of tobacco powder> to be described below. Examples of bases to be added include potassium carbonate and/or sodium carbonate, and it is preferable to add the base as an aqueous solution. In addition, a pH adjusting agent such as sodium dihydrogen phosphate can be added. The pH of the mixture after the base is added is preferably adjusted to 8.0 to 9.0.

**[0060]** The content of the tobacco powder in the mixture can be 60 to 90 weight%.

**[0061]** After the base is added, heating is performed under conditions of, for example, a product temperature of 65°C to 90°C and preferably a product temperature of 70°C to 80°C, and for example, for 0.5 to 3 hours and preferably for 0.8 to 2 hours. Thereby, the tobacco powder can be sterilized and the enzyme responsible for browning contained in the tobacco powder to be described below can be inactivated.

**[0062]** The heating can be performed by either or both of heating by steam injection and heating by a jacket.

**[0063]** The pH of the mixture after heating is preferably 8.0 to 9.0, and the water content of the mixture after heating is preferably 10 to 50 weight%.

**[0064]** Here, the method for measuring the pH of the mixture can be performed by the same procedure and device as in the method for measuring the pH of the tobacco filler described above.

**[0065]** The above heat treatment is called an alkaline sterilization treatment.

**[0066]** After heating, as necessary, steam injection into the obtained treated tobacco powder is stopped, only the

jacket is heated, and a drying treatment is performed.

**[0067]** Then, a mode in which cooling is performed at about 15°C to 25°C for about 1 hour can be exemplified.

**[0068]** Here, in the first mode, the tobacco powder subjected to extraction preferably exhibits a green color having an  $a^*$  value of 1.0 or less expressed by the CIELab color system method. When the tobacco powder has such a green color and the tobacco powder is heated in the presence of the above base, the bright green color of the tobacco powder is maintained. Then, when extraction is performed on the tobacco powder that retains its bright green color, the green pigment is also contained in the extraction liquid in addition to the solvent-soluble solids contained in the tobacco powder. When the extraction liquid is added to the packaging material, the dried packaging material exhibits a bright green color.

**[0069]** The shape and particle size of the tobacco powder subjected to extraction in the first mode are not particularly limited, but in order to increase an area in contact with the solvent, a small particle size is preferable.

**[0070]** The extraction residue obtained after extraction is performed on the tobacco powder can be mixed with the tobacco filler of the oral tobacco product. When the extraction residue is mixed with the tobacco filler, it is possible to adjust the content of the flavor component of the tobacco filler. It is possible to appropriately adjust the amount mixed in.

**[0071]** Regarding the second mode ((2) in Fig. 1), a method in which a slurry mixture containing a tobacco powder having an average particle size of preferably 30  $\mu\text{m}$  or less and a liquid dispersion medium is added as a tobacco material to a packaging material can be exemplified. Regarding the packaging material, a non-woven fabric can be exemplified.

**[0072]** Regarding the method for obtaining a tobacco powder used in the second mode, the following method can be exemplified.

**[0073]** The tobacco leaves are dried and then are preferably subjected to the alkaline sterilization treatment described in the first mode and dried, and then first coarsely crushed in a coarse crushing machine. While use of the tobacco powder subjected to the alkaline sterilization treatment is illustrated in Fig. 1, the present invention is not limited thereto.

**[0074]** The coarse crushing machine used in the coarse crushing step is not particularly limited. According to the coarse crushing step, it is possible to obtain a coarsely crushed tobacco powder having an average particle size of several hundreds of  $\mu\text{m}$  to several mm.

**[0075]** Next, a liquid dispersion medium is added to the coarsely crushed tobacco powder and the mixture is stirred and mixed.

**[0076]** Then, the mixture in which the coarsely crushed tobacco powder and liquid dispersion medium are stirred and mixed is subjected to a step of performing fine crushing using a wet fine crushing machine (for example, MIC-2: commercially available from Nara Machinery Co., Ltd.). The rotation speed of the machine is generally 1,100 to 1,300 rpm, and the fine crushing time is about 5 to 100 minutes.

**[0077]** The fine crushing step is performed and thus the tobacco powder is finely crushed, and the average particle size of the tobacco powder in the mixture is 30  $\mu\text{m}$  or less.

**[0078]** Here, in order to obtain a mixture containing a tobacco powder having an average particle size of 30  $\mu\text{m}$  or less and a liquid dispersion medium, a method using a dry fine crushing machine can be used in place of a method using the above wet fine crushing machine. Specifically, a method in which the coarsely crushed tobacco powder described above is finely crushed to obtain an average particle size of 30  $\mu\text{m}$  or less using a dry fine crushing machine such as a jet mill and a liquid dispersion medium is then added and the mixture is stirred and mixed can be exemplified.

**[0079]** Here, the average particle size of the tobacco powder in this specification is obtained by a laser diffraction and scattering method, a laser diffraction particle size distribution measurement device (for example, Shimadzu nanoparticle size distribution measurement device SALD-2100) is used as the device, and a refractive index is set to be within a range of 1.60 to 0.10i.

**[0080]** Using such a measurement principle and measurement device, the average value of the particle size obtained by analysis software bundled in the measurement device is set as an average particle size.

**[0081]** When the average particle size of the tobacco powder in the mixture containing the tobacco powder and the liquid dispersion medium is set to 30  $\mu\text{m}$  or less, in the mixture containing the tobacco powder and the liquid dispersion medium, the tobacco powder is uniformly and easily dispersed in the liquid dispersion medium. Further, regardless of the amount of the content of solids, the solids can be added to the packaging material so that the distribution of solids becomes uniform.

**[0082]** A mode in which the lower limit value of the average particle size of the tobacco powder is generally 5  $\mu\text{m}$  or greater, or 8  $\mu\text{m}$  or greater can be exemplified.

**[0083]** In order to increase the average particle size of the tobacco powder, when the fine crushing machine is used, it can be accomplished by shortening the fine crushing time or by adjusting the liquid dispersion medium at a low viscosity.

**[0084]** Regarding the liquid dispersion medium, one or more selected from the group consisting of water, monohydric alcohols, polyhydric alcohols, sugar alcohols, sugars and polyhydric alcohol esters can be selected.

**[0085]** The average particle size of the tobacco powder can be adjusted to a desired size using such a liquid dispersion medium.

**[0086]** Monohydric aliphatic alcohols such as methanol, ethanol, 1-propanol, 2-propanol, 1-butanol, 2-butanol, 2-methyl-1-propanol, 2,2-dimethylethanol, and cyclohexanol, monohydric alcohols having an aromatic substituent such

as benzyl alcohol, and additionally monohydric alcohols containing one or more halogen elements, and monohydric alcohols having one or more ether bonds can be exemplified.

**[0087]** The polyhydric alcohol in the present invention is a general term for compounds having two or more hydroxyl groups in one molecule, and the type thereof is not particularly limited. For example, glycerin and propylene glycol are preferably exemplified. Examples of sugar alcohols include sorbitol, maltitol, xylitol, erythritol, lactitol, sorbitan, xylose, arabinose, mannose, and trehalose. Examples of sugars include lactose, table sugar, coupling sugar, glucose, enzyme starch syrup, acid saccharified starch syrup, maltose starch syrup, maltose, isomerized sugar, fructose, reduced maltose, reduced starch starch syrup, and honey.

**[0088]** Examples of polyhydric alcohol esters include fatty acid polyhydric alcohol esters. Examples of fatty acid polyhydric alcohol esters include fatty acid triglycerides.

**[0089]** Among the above examples, water alone, propylene glycol alone, glycerin alone, or two or more thereof can be used in combination.

**[0090]** Among these, a combination of water and glycerin, a combination of water and propylene glycol, or propylene glycol alone is preferably used in order to adjust the average particle size to a desired range when the tobacco powder is finely crushed.

**[0091]** There is no particular limitation on a mixing ratio of water and the other dispersion medium, and in the case of water and glycerin, and water and propylene glycol, they can be mixed at an arbitrary ratio.

**[0092]** In the second mode, the mixture containing the tobacco powder and the liquid dispersion medium is in slurry form.

**[0093]** In order to exhibit such a property of being in slurry form, the weight ratio between the liquid dispersion medium and the tobacco powder can be generally 1.5 to 99 (the weight of the tobacco powder is 1 to 40 weight% with respect to the total amount of the mixture) when the weight of the tobacco powder is set as 1.

**[0094]** Since the mixture containing the tobacco powder and the liquid dispersion medium exhibits slurry, when it is sprayed on the packaging material using, for example, a spray, the mixture containing the tobacco powder and the liquid dispersion medium can be uniformly evenly added to the packaging material. Thereby, solids derived from the tobacco powder can be uniformly added to the packaging material. In addition to this, a method of immersing a packaging material to the mixture containing the tobacco powder and the liquid dispersion medium and adding the mixture to the packaging material by printing can be exemplified.

**[0095]** The mixture obtained above is added so that the weight of solids in the packaging material is 11.0 g/m<sup>2</sup> or greater, preferably 12.0 g/m<sup>2</sup> or greater, and more preferably 15.0 g/m<sup>2</sup> or greater.

**[0096]** Here, as in the first mode, after the above mixture is added, the packaging material is dried, and thus the liquid dispersion medium constituting the mixture is removed. Thereby, solids contained or dissolved in the mixture are deposited inside and on the surface of the packaging material. The above "weight of solids of 11.0 g/m<sup>2</sup> or greater" is based on the weight after drying.

**[0097]** Here, regarding the upper limit value of the weight of solids added to the packaging material, a mode in which addition is performed so that the weight of solids is, for example, 40.0 g/m<sup>2</sup> or less can be exemplified and a mode in which addition is performed so that the weight of solids is 35.0 g/m<sup>2</sup> or less can be exemplified.

**[0098]** The third mode ((3) in Fig. 1) is a method for adding a tobacco powder to a packaging material without using a dispersion medium or a solvent.

**[0099]** For example, when the packaging material is manufactured, the packaging material can be produced by mixing a packaging raw material and a tobacco powder in advance.

**[0100]** For example, a method in which, when the packaging material is a non-woven fabric, during papermaking of a non-woven fabric, a tobacco powder is mixed with wood pulp which is a non-woven fabric raw material, papermaking is then performed and thus the tobacco material is incorporated into the non-woven fabric can be exemplified.

**[0101]** Either wet papermaking or dry papermaking can be used during papermaking of a non-woven fabric, and when dry papermaking is used, a tobacco powder can be easily mixed in during papermaking.

**[0102]** Regarding the tobacco powder, those having an average particle size of 30 μm or less can be used. Tobacco powders having other average particle sizes can be used. A mode in which the lower limit value of the average particle size of the tobacco powder is generally 5 μm or greater or 8 μm or greater can be exemplified. Regarding a method for preparing a tobacco powder having an average particle size of 30 μm or less, the method described in the second mode can be used. In addition, regarding the average particle size measurement method, the method described in the second mode can be used. Regarding the tobacco powder, those subjected to the alkaline sterilization treatment described in the first mode are preferably used. While use of the tobacco powder subjected to the alkaline sterilization treatment is illustrated in Fig. 1, the present invention is not limited thereto.

**[0103]** Regarding fibers constituting the non-woven fabric, either synthetic fibers or natural fibers can be used. Cellulose fibers can be exemplified as an examples of natural fibers. The basis weight of the non-woven fabric can be about 10 to 50 g/m<sup>2</sup> and is preferably 15 to 40 g/m<sup>2</sup>.

**[0104]** The conditions for this non-woven fabric can also be used when a non-woven fabric is used as a packaging material not only in the third mode but also in the first mode and the second mode.

**[0105]** When the tobacco powder used in each of the above modes of the packaging material has a green color having an  $a^*$  value of 1.0 or less expressed by the CIELab color system method, in any of the above modes, a bright green tobacco powder as a solid is exposed outside of the packaging material, and an oral tobacco product having a favorable appearance is obtained.

**[0106]** In order to secure a green color having an  $a^*$  value of 1.0 or less expressed by the CIELab color system method for the tobacco powder, those obtained through the alkaline sterilization treatment described above are preferable.

#### <Manufacture of tobacco product>

**[0107]** The tobacco product according to the embodiment of the present invention can be obtained by packaging a tobacco filler with a packaging material containing the above tobacco material and performing sealing by a known method ((4) in Fig. 2). Regarding the composition of the tobacco filler, those described in the section <Tobacco product> can be used. When the packaging material is a non-woven fabric, for example, sealing can be performed by a method such as heat sealing. The sealed package is also called a pouch.

**[0108]** Since the packaging material used in the embodiment of the present invention is liquid-permeable, when a solvent containing ethanol and/or propylene glycol is added to the package obtained by filling a tobacco filler with a packaging material from the outside, the solvent reaches the tobacco filler which is the content of the package. Then, the solvent dissolves a solvent-soluble substance contained in the tobacco filler and then the solvent-soluble substance exudes outside of the packaging material together with the solvent. Propylene glycol is preferably used as the solvent.

**[0109]** When the tobacco powder used as a filler of the tobacco product has a green color having an  $a^*$  value of 1.0 or less expressed by the CIELab color system method, after the tobacco filler is filled into the packaging material, a solvent containing ethanol and/or propylene glycol is added from the outside, and thus a pigment contained in the tobacco filler also exudes outside of the packaging material. Then, the appearance of the tobacco product can be changed to bright green. Preparation of the tobacco powder having a green color will be described below.

**[0110]** When the tobacco product according to the embodiment of the present invention is manufactured, as in the three modes described in the above section of the method for manufacturing a packaging material, it is possible to use a packaging material into which a tobacco material is incorporated in advance by any method with respect to the packaging material ((4) in Fig. 2). In addition, when the tobacco product according to the embodiment of the present invention is manufactured, after a package in which a tobacco filler is packaged is obtained using a packaging material into which a tobacco material is not incorporated in advance, a solvent containing ethanol and/or propylene glycol is added from the outside, a solvent-soluble substance contained in the tobacco filler exudes from outside of the package, and solids of the tobacco material can be incorporated into the packaging material of the tobacco product which is a desired final product ((5) in Fig. 2). When the weight of the tobacco filler is set to 1, the weight of the solvent containing ethanol and/or propylene glycol added from the outside is preferably 0.2 to 2.0 and more preferably 0.3 to 0.7 because this enables the solvent-soluble substance to be efficiently exuded from the tobacco filler. The solvent added from the outside can be, for example, ethanol alone, propylene glycol alone, or a mixture thereof. A very small amount of water can be mixed into these. Among these, ethanol alone or propylene glycol alone is preferably used. After the solvent is added, exudation of the solvent-soluble substance from the tobacco filler is confirmed, and drying is then performed, and the solvent is preferably removed.

#### <Preparation of tobacco powder>

**[0111]** Regarding the tobacco powder used in the embodiment of the present invention, it is preferable that both one used for the tobacco filler and one used for a tobacco material (the tobacco powder contained in the packaging material) used when a packaging material is manufactured have a green color having an  $a^*$  value of 1.0 or less expressed by the CIELab color system method. In order to make the green color stronger, an  $a^*$  value of -2.0 or less is preferable. On the other hand, an  $a^*$  value of -20 or greater is appropriate in order to secure the green color of the tobacco powder.

**[0112]** The  $a^*$  value of the tobacco powder can be measured using an optical colorimeter (for example, KONICA MINOLTA/ CM3500d, Konica Minolta Holdings, Inc.) after drying is performed until the water content is 3 to 5 weight%. Here, the definition of color is expressed by the  $L^*a^*b^*$  color system adopted by Commission International de l'Eclairage (CIE) and JIS.

**[0113]** A color measurement operation is performed by putting a sample tobacco powder into a glass container with a layer thickness of 1 cm, applying standard light (color measurement standard Illuminant D65, CIE, ISO reference light) from the bottom of the container, and measuring reflected light (reflective color measurement/specular component excluded method (SCE)), and the measured value is accordingly digitized.

**[0114]** The color measurement of the tobacco filler and the packaging material itself can be performed according to the above operation.

**[0115]** In addition, regarding the tobacco powder and the tobacco filler used in the embodiment of the present invention,

the activity value of the enzyme responsible for browning (polyphenol oxidase: PPO) is preferably 0.1 U/g or less.

**[0116]** The fact that the activity value of the enzyme responsible for browning contained in the tobacco powder is 0.1 U/g or less means that the enzyme responsible for browning the tobacco powder and the tobacco filler is inactivated, and in this case, the green color of the tobacco powder and the tobacco filler is maintained during storage. The definition of U will be described below.

**[0117]** Since the enzyme responsible for browning functions after harvesting of tobacco leaves for the tobacco powder raw material and turns the tobacco leaves yellow, in order to obtain the above green color and maintain the green state, it is preferable to perform an operation of inactivating the enzyme responsible for browning as soon as possible after harvesting of the tobacco leaves.

**[0118]** Here, the activity value of the enzyme responsible for browning of general tobacco leaves (Burley species) is about 4.5 to 6.5 U/g.

**[0119]** Here, "turn yellow" means that most of the area of the harvested tobacco leaves, for example, 60% or greater, for example, 90% or greater, is discolored until the  $a^*$  value becomes a value larger than 1.0. The tobacco leaves turn yellow due to a decrease in the (green) pigment present in the tobacco leaves after harvesting. Here, the  $a^*$  value after harvesting of tobacco leaves is generally about -9 to -1.5. For example, for general tobacco leaves, Burley species, yellow species, and *Nicotiana rustica*, the  $a^*$  value of the tobacco leaves (frozen product) before drying after harvesting is  $-2.1 \pm 0.3$  for Burley species,  $-2.5 \pm 0.6$  for yellow species, and  $-4.6 \pm 0.5$  for *Nicotiana rustica*.

**[0120]** Regarding the tobacco leaves used for preparing the tobacco powder, those having an  $a^*$  value of 1.0 or less and a water content of 7 weight% or less are preferably used. Tobacco leaves that are harvested earlier than a harvest time for tobacco leaves that are used for general cigarettes are greener than general leaves, and thus such tobacco leaves are preferably used. For measurement of the  $a^*$  value of tobacco leaves, 20 points on the surface of tobacco leaves are set as measurement targets, and color measurement can be performed using the above spectral colorimeter. 20 points on the surface of tobacco leaves are evenly selected from the center and the outer circumferential part of the tobacco leaves (for example, refer to Fig. 4 in WO 2016/043160).

**[0121]** Such tobacco leaves can be obtained by performing a known drying treatment before turning yellow, but they can also be obtained by performing the following drying treatment before turning yellow ("dry" in Fig. 3).

**[0122]** First, a mode in which drying is performed at 20°C to 40°C and a relative humidity (can be expressed as a difference between "dry bulb temperature" and "wet bulb temperature") of 40% RH to 80% RH (hereinafter "% RH" will be used as a unit of relative humidity) for 3 to 24 hours (fermentation stage), drying is then performed at 40°C to 55°C and a relative humidity of 40% RH to 70% RH for 24 to 72 hours (color fixing stage: leaves are dried), and drying is then performed at 60°C to 70°C and a relative humidity of 5% RH to 30% RH for 0 to 120 hours (stem dry stage: dry up to stems) can be exemplified. When this method is used, respective steps can be performed separately or can be performed continuously. In order to maintain the green color of tobacco leaves, it is preferable to perform the steps continuously.

**[0123]** In addition, drying in the stem dry stage can be omitted, and in this case, the drying time in the color fixing stage is lengthened and the water content of the obtained tobacco leaves is sufficiently reduced to at least 7 weight% or less. A known device can be used for drying in the above temperature range, and a constant temperature and constant humidity dryer or a hot air dryer can be used.

**[0124]** When the content of water contained in tobacco leaves exceeds 7 weight%, the green color of the tobacco leaves is significantly reduced in the subsequent heating step.

**[0125]** After harvesting, those obtained by removing stems from tobacco leaves can be subjected to the above drying treatment or after harvesting, tobacco leaves can be subjected to the above drying treatment after removing water by squeezing. In addition, after harvesting, the tobacco leaves can be refrigerated or frozen-stored and then subjected to the above drying treatment.

**[0126]** After the above drying treatment, tobacco leaves can be subjected to a predetermined heat treatment to inactivate the enzyme responsible for browning contained in the tobacco leaves. Regarding a method for reducing the activity value of the enzyme responsible for browning of the tobacco powder without performing this heat treatment, an alkaline sterilization treatment to be described below can be exemplified.

**[0127]** The lower limit of the heating temperature here is 75°C or higher, and can be 80°C or higher in another mode, and 85°C or higher in still another mode. On the other hand, the upper limit of the heating temperature can be 99°C or lower, and 90°C or lower in another mode. The heating temperature can be kept almost constant at a temperature within the above range in the heat treatment.

**[0128]** In addition, the relative humidity during heating can be 3% RH to 60% RH.

**[0129]** In addition, the relative humidity during heating may be kept almost constant within the above range of the relative humidity.

**[0130]** Here, being kept almost constant means a mode in which the relative temperature and/or relative humidity is maintained continuously with a variation width of about  $\pm 10\%$ .

**[0131]** In addition, the heating time can be about 1 hour to 3 days.

**[0132]** Regarding the same heating temperature conditions, a mode in which a heating time is lengthened when the

relative humidity is lowered (for example, when it is 20% RH or less) in order to reliably inactivate the activity of the enzyme responsible for browning and a heating time is shortened when the relative humidity is increased (for example, when it exceeds 30% RH) in order to keep the  $a^*$  value low can be exemplified.

[0133] Specific modes of the heating temperature, relative humidity, and heating time can be exemplified as follow.

[0134] When the heating temperature is set to 75°C or higher and lower than 80°C and the relative humidity is set to 3% RH or greater and 20% RH or less, the heating time can be 48 hours or longer. On the other hand, the upper limit of the heating time in this case can be 96 hours or shorter.

[0135] When the heating temperature is set to about 80°C to 85°C and the relative humidity is set to 3% RH or greater and less than 10% RH, the heating time can be 24 hours or longer. On the other hand, the upper limit of the heating time in this case can be 96 hours or shorter.

[0136] When the heating temperature is set to about 80°C to 85°C and the relative humidity is set to 10% RH or greater and less than 40% RH, the heating time can be about 4 to 18 hours.

[0137] When the heating temperature is set to about 80°C to 85°C and the relative humidity is set to 40% RH or greater and 60% RH or less, the heating time can be about 1 to 2 hours.

[0138] In addition to the above modes, any mode can be used without limitation as long as when the  $a^*$  value of the treated tobacco leaves is 1.0 or less and the activity value of the enzyme responsible for browning is 0.1 U/g or less.

[0139] Regarding a device that can be used during heating such that the  $a^*$  value of the treated tobacco leaves is 1.0 or less and the activity value of the enzyme responsible for browning is 0.1 U/g or less, a constant temperature and humidity chamber can be exemplified. Specifically, a constant temperature and humidity chamber (PR-3KPH, commercially available from ESPEC CORP.) can be exemplified.

[0140] Here, the activity value of the enzyme responsible for browning of tobacco leaves or tobacco powder can be determined by adding a solution obtained by extracting an enzyme protein from a sample and a potassium phosphate buffer (pH 6.0) to a cell of a spectral absorption spectrophotometer and performing mixing, adding a 10 mM pyrocatechol solution as a substrate thereto, and measuring the increase in the absorbance at a wavelength of 420 nm with respect to a reference at 40°C. Regarding the reference, instead of an enzyme protein solution, a solution in which a potassium phosphate buffer is mixed can be exemplified. 1 U is defined as the amount of the enzyme that increases the absorbance ( $\Delta$ ABS) of the sample by 0.01 per minute by subtracting the increase in the absorbance of the reference.

[0141] Here, the extraction conditions for tobacco leaves or tobacco powder can include the following. 1 g of a crushed tobacco leave sample or tobacco powder is weighed out into a 100 ml vial, 50 ml of a 20 mM potassium phosphate buffer (pH 6.0) is added thereto, and the mixture is homogenized under an ice-cold environment for 2 minutes, and additionally ultrasonicated under an ice-cold environment for 30 minutes.

[0142] Then, the extract is filtered using a 0.2  $\mu$ m membrane filter (membrane material: cellulose acetate). This filtrate is used as a crude enzyme protein solution for measuring enzyme activity.

[0143] After the above drying treatment or after the above drying treatment and heat treatment, a treatment for removing stems from tobacco leaves is performed by a known method to obtain a lamina ("stem separation" in Fig. 3), and additionally crushing is then performed ("crush" in Fig. 3) to obtain a tobacco powder. When the water content of tobacco leaves is kept at 7 weight% or less during the period (during treatments surrounded by dotted lines in Fig. 3) from the above drying treatment (and the heat treatment as necessary) to the stem separation treatment and the crushing treatment until a tobacco powder is obtained, this contributes to maintaining the green color of the tobacco powder. In order to keep the water content of tobacco leaves at 7 weight% or less, for example, during transportation, it is preferable to store tobacco leaves in a water impermeable packaging material.

<Preparation of tobacco filler>

[0144] The tobacco powder obtained through the above treatments is mixed with a salt and a base and, as necessary, a filling material and water (Fig. 4). The content of the base is adjusted so that the pH of the mixture becomes alkaline. When the tobacco powder obtained through the above treatments has a green color having an  $a^*$  value of 1.0 or less in the CIELab color system, the tobacco powder that retains its green color even after the following operation is obtained.

[0145] Regarding the filling material, those described in the section <Tobacco product> can be used.

[0146] Examples of salts can include one or more selected from the group consisting of sodium chloride, potassium chloride, sodium citrate, potassium citrate, sodium acetate, and potassium acetate. Among these, at least sodium chloride is preferably used.

[0147] Examples of bases include one or more selected from the group consisting of potassium carbonate, sodium carbonate, and sodium bicarbonate. Among these, at least sodium carbonate and/or potassium carbonate is preferably used. For the base, an aqueous solution can be prepared and used.

[0148] As a specific procedure, first, a filling material and a salt are added to and mixed with a tobacco powder to prepare a mixture, and an aqueous solution in which a base is dissolved is then added to the mixture. The base aqueous solution is added to the mixture using a sprayer or the like, and thus the base can be uniformly added. In addition, as

necessary, a pH adjusting agent such as sodium dihydrogen phosphate can be added to the mixture.

**[0149]** The pH of the mixture containing at least the tobacco powder, salt, and base obtained through this operation is 8.3 or greater and preferably 8.4 or greater.  $8.5 \pm 0.1$  is more preferable. The pH can be measured according to the pH measurement method for the tobacco filler described above.

**[0150]** The content (dry weight) of the tobacco powder in the dry weight of the mixture can be 35 to 90 weight% or 45 to 60 weight%.

**[0151]** The water content of the mixture before and after heating is about 5 to 50 weight% and is preferably 10 to 45 weight%.

**[0152]** The content of the salt in the dry weight of the mixture can be, for example, 0.1 to 12 weight%, and is preferably 3.0 to 8.0 weight%.

**[0153]** The content of the base in the dry weight of the mixture can be, for example, 0.1 to 12 weight%, and is preferably 5.0 to 9.0 weight%.

**[0154]** The above mixture is subjected to a heat treatment for sterilization. The heat treatment is a treatment in which the above mixture is heated at a temperature for a time sufficient for low temperature sterilization. The heating temperature can be, for example, about 65°C to about 90°C for the product temperature, and is preferably about 70°C to 80°C for the product temperature.

**[0155]** The heating time of the mixture is not particularly limited, and can be generally about 1 hour to about 3 hours, and at least about 1 hour. Examples of heating methods include one or both of increasing a jacket temperature of a mixer holding the mixture (jacket heat) and injecting steam into the mixture.

**[0156]** For example, heating can be performed by steam injection first, and jacket heating may be then performed.

**[0157]** During this heat treatment, the pH of the above mixture is preferably maintained at 8 or greater, more preferably maintained at 8.3 or greater, and particularly preferably maintained at about  $8.5 \pm 0.1$ .

**[0158]** The enzyme responsible for browning contained in the tobacco powder is inactivated when this heat treatment is performed. Therefore, the activity value of the enzyme responsible for browning of the tobacco filler obtained through this heat treatment is 0.1 U/g or less. This heat treatment is also called an alkaline sterilization treatment. The difference from the alkaline sterilization treatment performed during preparation of a tobacco powder used for preparation of a tobacco powder extraction liquid is that a filling material and a salt are added to the mixture to be treated.

**[0159]** After the above heat treatment, a treatment of drying the above mixture can be performed. Then, a cooling treatment is performed. The cooling can be natural cooling or can be performed using any cooling method. When drying is performed, for example, the water content of the above mixture can be adjusted to 5 to 45 weight%. Thereby, it is easy to adjust the water content in the tobacco filler as a desired product.

**[0160]** After cooling, an additive can be added to the above mixture. Examples of additives include a sweetener, a bitterness inhibitor, a flavor material, a pH adjusting agent, and a humectant.

**[0161]** Regarding the amount of the additive added, the amount of the sweetener can be 0.1 to 1.0 weight% with respect to the total weight of the tobacco filler, the amount of the flavor material can be 0.1 to 5.0 weight% with respect to the total weight of the tobacco filler, the amount of the pH adjusting agent can be 0.1 to 10.0 weight% with respect to the total weight of the tobacco filler, and the amount of the humectant can be 0.1 to 10.0 weight% with respect to the total weight of the tobacco filler. Here, the pH adjusting agent can be added before the heat treatment and in this case, it is not required to add after the heat treatment.

**[0162]** When the above additive is added as necessary, it is possible to obtain the tobacco filler according to the embodiment of the present invention. Here, while "filling material" is shown in Fig. 4, this is an optional component. In addition, while "cool and dry" and "add additive" are shown in Fig. 4, these are optional treatments, and can be appropriately changed.

**[0163]** The oral tobacco product of the present invention can quickly deliver the characteristic flavor and taste of the tobacco material during use. In addition, when the tobacco material contained in the packaging material has a specific green color, a refreshing impression can be provided to the user.

**[0164]** For a simple description of embodiments of the tobacco product of the present invention, for example, the following modes can be exemplified. In any of the modes, the weight of solids in the packaging material is 11.0 g/m<sup>2</sup> or greater, more preferably 12.0 g/m<sup>2</sup> or greater, and still more preferably 15.0 g/m<sup>2</sup> or greater. On the other hand, the weight of solids in the packaging material can be 40.0 g/m<sup>2</sup> or less.

**[0165]** First, a mode of an oral tobacco product in which a tobacco filler is filled into a packaging material of which a part or all of the packaging material has a green color having an a\* value of 1.0 or less, and for the tobacco material, the tobacco powder extraction liquid is added ((1) in Fig. 1), the mixture of tobacco powder and a liquid dispersion medium is added ((2) in Fig. 1), and addition is performed during manufacturing of the packaging material ((3) in Fig. 1: when the packaging material is a non-woven fabric, for example, mixing during papermaking) can be exemplified. These are obtained according to the manufacturing method of (4) in Fig. 2. In such a mode, when the tobacco powder used for manufacturing the packaging material has the green color described above, the tobacco product has a bright green appearance. A part or all of the surface of the packaging material can be green. In such a mode, the color of the tobacco

filler can be or is not necessarily the green color described above.

**[0166]** Therefore, regarding the tobacco filler used in such a mode, those subjected to the alkaline sterilization treatment shown in Fig. 4 can be preferably used, but the tobacco filler is not necessarily limited thereto.

**[0167]** When the color of the tobacco filler is also the green color described above and the activity value of the enzyme responsible for browning is 0.1 U/g or less, it is possible to prevent the inside of the oral cavity of the user from becoming brown when the oral tobacco product is used.

**[0168]** In addition, regarding an oral tobacco product, a product in which a tobacco filler is filled into a packaging material and sealing is performed and a solvent containing ethanol and/or propylene glycol is then added from the outside, and a solvent-soluble substance contained in the tobacco filler exudes outside of the packaging material can be exemplified ((5) in Fig. 2). In this mode, the packaging material has a solid content of 11.0 g/m<sup>2</sup> or greater. In addition, in this mode, when the tobacco filler that is contained and has the green color described above, since a pigment is also contained in the component that exudes outside of the packaging material, the tobacco product has a bright green appearance. A part or all of the surface of the packaging material can be green. In addition, in such a mode, when the activity value of the enzyme responsible for browning of the tobacco filler is 0.1 U/g or less, it is possible to prevent the inside of the oral cavity of the user from becoming brown when the oral tobacco product is used.

**[0169]** In any of the above modes, when the tobacco powder and/or tobacco filler having a green color having an a\* value of 1.0 or less is used, the content shown in Fig. 3 is preferably performed as treatments after harvesting of tobacco leaves. In addition, in the subsequent preparation of the tobacco filler, the alkaline sterilization treatment shown in Fig. 4 is preferably performed.

**[0170]** In addition, preparation of the tobacco powder and preparation of the tobacco filler shown in Fig. 3 and Fig. 4 can be performed successively. In addition, a method for manufacturing a tobacco product including all of a series of steps, including the subsequent step of manufacturing a tobacco product, can be used. In this case, both the tobacco material contained in the packaging material of the obtained tobacco product and the tobacco filler have a green color having an a\* value of 1.0 or less. In addition, the activity value of the enzyme responsible for browning of both the tobacco material and the tobacco filler is 0.1 U/g or less.

[Examples]

**[0171]** While the present invention will be described in more detail with reference to examples, the present invention is not limited to the descriptions of the following examples without departing from the spirit and scope.

<Preparation of tobacco filler>

**[0172]** A Burley species (2017 domestically harvested earlier than a general harvest time) was selected as tobacco leaves.

**[0173]** After harvesting and before turning yellow, drying was performed under an atmospheric pressure while gradually increasing the temperature from 36°C to 72°C for 73 hours, and the water content was reduced to 5 weight%.

**[0174]** The dried tobacco leaves were subjected to a stem separation treatment and a crushing treatment to prepare 3 kg of a tobacco powder. 2 kg of a cellulose powder as a filling material and 317 g of sodium chloride as a salt were added thereto and mixed. Then, an aqueous solution in which potassium carbonate (189 g) and sodium carbonate (196 g) were mixed was sprayed as a base. In addition, a sodium dihydrogen phosphate (241 g) aqueous solution was sprayed and mixed until uniform. The pH of the obtained mixture was 8.5.

(Alkaline sterilization treatment)

**[0175]** The obtained mixture was directly heated with steam for 10 minutes (the pH after heating was 8.8), and jacket heating was then performed for 1 hour so that the raw material product temperature was 70°C to 80°C (the pH after heating was 8.5).

**[0176]** Then, cooling was performed under conditions of an ambient temperature of 20°C for 1 hour (the pH after cooling was 8.5).

**[0177]** A flavor material was sprayed on the cooled mixture. Finally, the pH was adjusted to 8.5 and a tobacco filler was produced.

<Preparation of tobacco powder used for producing extraction liquid>

**[0178]** The above tobacco leaves dried after the harvesting were subjected to a stem separation treatment and a crushing treatment to prepare 5 kg of a tobacco powder (dry weight 4,773 g). An aqueous solution in which potassium carbonate (315 g; 6.6% with respect to the dry tobacco powder) and sodium carbonate (198.1 g; 4.15% with respect to

the dry tobacco powder) were mixed was sprayed as a base to the tobacco powder. In addition, a sodium dihydrogen phosphate (242.5 g; 5.08% with respect to the dry tobacco powder) aqueous solution was sprayed and mixed until uniform, and thus the pH of the mixture was adjusted to 8.7.

5 (Alkaline sterilization treatment)

[0179] The obtained mixture was directly heated with steam for 10 minutes (the pH after heating was 8.9), and jacket heating (can wall temperature of 100°C) was then performed for 1 hour so that the raw material product temperature was 70°C to 80°C (the water content after heating was 12.6 weight%, and the pH was 8.7 (measured by the same measurement method as the tobacco filler described above)). Here, in this case, the target value of the pH was 8.5 to 9.0, and the water content was 10 to 15 weight%.

[0180] Then, cooling was performed under conditions of an ambient temperature of 20°C for 1 hour (the pH after cooling was 8.7).

15 <Manufacture of oral tobacco product>

[0181]

20 (1) A non-woven fabric (commercially available from Tenowo, basis weight 34.5 g/m<sup>2</sup>) was filled with the above tobacco filler to obtain 0.6 g/piece, and sealing was performed by heat sealing to produce an oral tobacco product of Comparative Example 1.

The weight of solids in the packaging material was 10.5 g/m<sup>2</sup>.

25 (2) 100% ethanol was added to a ready-made snus pouch and the pouch was then sealed. The amount of ethanol sprayed was adjusted to 4.0 g for 20 snus pouches. Thereby, the ethanol-soluble substance contained in the tobacco filler was leaked outside of the packaging material. This was used as a tobacco product of Example 1. The amount of the tobacco filler of the ready-made snus pouch was 0.35 g/piece.

In addition, the weight of solids in the packaging material of the tobacco product of Example 1 was 20.0 g/m<sup>2</sup>.

30 (3) 50 g of the tobacco powder prepared in the above <Preparation of tobacco powder used for producing extraction liquid> was dispersed in 100 g of ethanol and stirred at 22°C for 14 hours, and thus an extraction operation was performed. 34.5 g/m<sup>2</sup> of a non-woven fabric was immersed for 1 second in an extraction liquid in which a tobacco powder having a large particle size was separated using a mesh and the non-woven fabric was then dried for 10 minutes to obtain a packaging material. The above tobacco filler was packaged at 0.35 g/piece using the packaging material, and heat-sealed to produce a tobacco product of Example 2.

35 [0182] The weight of solids in the packaging material of the tobacco product of Example 2 was 28.5 g/m<sup>2</sup>. When the color of the packaging material was measured (Color Reader CR-20 commercially available from KONICA MINOLTA), the a\* value was -5.9.

[0183] The weight of solids contained in the above packaging materials (1) to (3) was measured according to the procedures described above.

40

<Sensory evaluation>

[0184] The following sensory evaluation was performed using the above tobacco products.

Evaluation content: status in which flavor and taste rose when the tobacco product was in the mouth

45 [0185] Impact on the characteristic flavor, taste, and sensation moving from the oral cavity to the nasal cavity of the tobacco material felt by user in mouth for first 15 seconds.

(Evaluation method)

50 [0186] The oral tobacco products after storage were evaluated by 11 general panelists for snus according to sensory evaluation. The panelists put the oral tobacco product in their mouth for 15 seconds and then evaluated the flavor according to the following evaluation criteria. When the average score of 11 panelists was greater than 2 points (had a high feeling of rising), this was evaluated as "A," when the average score was 1 to 2 points (had a relatively high feeling of rising), this was evaluated as "B," and when the average score was less than 1 point (had a low feeling of rising), this was evaluated as "C." Here, the panelists took 5-minute breaks between evaluating products.

55

3: felt strongly

2: felt slightly

1: very slightly recognizable

0: not felt at all

**[0187]** The results are shown in Table 1 as a summary table, and a summary thereof is shown in Table 2. Based on the results in Table 2, it was shown that the product according to the embodiment of the present invention had better rising of the characteristic flavor and taste of the tobacco material than the conventional snus pouch.

[Table 1]

**[0188]**

Table 1 Summary table

Panelist name	Evaluation point(3,2,1,0)		
	Comparative Example 1	Example 1	Example 2
1	0	2	3
2	1	2	3
3	0	1	3
4	0	2	3
5	0	1	2
6	0	2	3
7	1	2	3
8	0	1	3
9	1	2	3
10	0	2	3
11	0	2	3
Sum	0.3	1.7	2.8
Determination	C	B	A

[Table 2]

**[0189]**

Table 2 Evaluation results

	Content	Average evaluation point	Determination
Comparative Example 1	Conventional product	0.3	C
Example 1	Product obtained by packaging tobacco tobacco filler with packaging material and then spraying 100% ethanol	1.7	B
Example 2	Product obtained by packaging tobacco tobacco filler with packaging material to which tobacco powder extract was added	2.8	A

**[0190]** A packaging material was produced according to the same operations as in Example 2 except that, in production of the packaging material of Example 2, the weight ratio between the tobacco powder and ethanol when the extraction liquid was produced was changed. As shown in Table 3, even if the proportion of ethanol was higher, sufficient solids can be added to the packaging material. In addition, the a\* value of the packaging material in this case was smaller than -5 and the material exhibited a bright green color. In addition, an oral tobacco product was produced using this packaging

material in the same manner as in the oral tobacco product of Example 2, and the above <Sensory evaluation> was performed, and in all cases, the results corresponding to A were obtained.

[Table 3]

[0191]

Table 3

	Tobacco powder:ethanol (weight ratio)	Solid content weight (g/m <sup>2</sup> )	a* value
Example 3	1:3	19.0	-5.4
Example 4	1:4	16.0	-6.6
Example 5	1:5	12.7	-5.1

### Claims

1. An oral tobacco product including, as constituent elements, a tobacco filler and a liquid-permeable packaging material for packaging the tobacco filler,  
wherein the packaging material includes solids derived from a tobacco material and a weight of the solids in the packaging material is 11.0 g/m<sup>2</sup> or greater.
2. The oral tobacco product according to claim 1,  
wherein the liquid-permeable packaging material includes a green part having an a\* value in a CIELab color system of 1.0 or less.
3. The oral tobacco product according to claim 1 or 2,  
wherein the liquid-permeable packaging material is composed of a non-woven fabric.
4. The oral tobacco product according to any one of claims 1 to 3,  
wherein the packaging material is obtained by adding thereto any of the following 1) to 3) as a tobacco material:
  - 1) a tobacco powder extraction liquid obtained using a solvent containing ethanol and/or propylene glycol as an extraction solvent;
  - 2) a slurry mixture containing a tobacco powder having an average particle size of 30 μm or less and a liquid dispersion medium; and
  - 3) a tobacco powder.
5. The oral tobacco product according to claim 4,  
wherein an a\* value of the tobacco powder is 1.0 or less, and an activity value of an enzyme responsible for browning of the tobacco powder is 0.1 U/g or less.
6. The oral tobacco product according to any one of claims 1 to 5,  
wherein the tobacco filler has a green color having an a\* value of 1.0 or less in a CIELab color system.
7. A method for manufacturing a packaging material for an oral tobacco product, the method comprising a step of adding a tobacco material to a liquid-permeable packaging material,  
wherein the step is a step of adding the tobacco material to the liquid-permeable packaging material so that a weight of solids in the packaging material is 11.0 g/m<sup>2</sup> or greater.
8. The method for manufacturing a packaging material for an oral tobacco product according to claim 7,  
wherein the liquid-permeable packaging material is a non-woven fabric.
9. The method for manufacturing a packaging material for an oral tobacco product according to claim 7 or 8, wherein the tobacco material is a slurry mixture containing a tobacco powder having an average particle size of 30 μm or less and a liquid dispersion medium.

10. The method for manufacturing a packaging material for an oral tobacco product according to claim 7 or 8, wherein an extraction liquid obtained by a step of extracting a tobacco powder using a solvent containing ethanol and/or propylene glycol as an extraction solvent is used as the tobacco material.

11. The method for manufacturing a packaging material for an oral tobacco product according to claim 10, wherein a weight ratio between the tobacco powder and the solvent in the extraction liquid is 1:1.5 to 1:8.

12. The method for manufacturing a packaging material for an oral tobacco product according to any one of claims 9 to 11, wherein the tobacco powder is obtained through an alkaline sterilization treatment.

13. The method for manufacturing a packaging material for an oral tobacco product according to any one of claims 9 to 12, wherein the step of adding the tobacco material to the liquid-permeable packaging material is performed by immersion, spraying, or printing.

14. The method for manufacturing a packaging material for an oral tobacco product according to claim 8, wherein the addition of the tobacco material is performed by adding a tobacco powder during papermaking of the non-woven fabric.

15. A method for manufacturing an oral tobacco product, the method comprising a step of filling the packaging material obtained by the method for manufacturing a packaging material for an oral tobacco product according to any one of claims 7 to 14 with a tobacco filler.

16. The method for manufacturing an oral tobacco product according to claim 15, wherein the following steps are included, as a step of manufacturing the tobacco filler, before the step of filling the packaging material with the tobacco filler:

- 1) a step of mixing a tobacco powder, a salt, and a base;
- 2) a step of heating the mixture obtained through the step 1);
- 3) a step of cooling and drying the mixture after heating; and
- 4) a step of adding an additive to the mixture after drying.

17. The method for manufacturing an oral tobacco product according to claim 16, wherein when the tobacco material added to the packaging material is an extraction liquid obtained by a step of extracting a tobacco powder using a solvent containing ethanol and/or propylene glycol as an extraction solvent, the method comprises a step of adding an extraction residue obtained in the extraction step to the tobacco filler.

18. The method for manufacturing an oral tobacco product according to claim 16 or 17, wherein the following steps a) and b) are included, as a step of manufacturing the tobacco powder used for manufacturing the tobacco filler, before manufacturing the tobacco filler, and a water content of the tobacco powder is kept at 7 weight% or less after the step a) until the step b) and the step 1):

- a) a step of drying tobacco leaves after harvesting the tobacco leaves until a water content of the tobacco leaves is 7 weight% or less; and
- b) a step of obtaining a tobacco powder by crushing the tobacco leaves after the step of drying.

19. A method for manufacturing an oral tobacco product, the method comprising:

- a step of filling a liquid-permeable packaging material with a tobacco filler having a green color having an a\* value of 1.0 or less in a CIELab color system; and
- a step of adding a solvent containing ethanol and/or propylene glycol to a package after the filling from outside of the package.

20. The method for manufacturing an oral tobacco product according to claim 19, wherein the following steps are included as a step of manufacturing the tobacco filler before the step of filling the packaging material with the tobacco filler:

- 1) a step of mixing a tobacco powder, a salt, and a base;
- 2) a step of heating the mixture obtained through the step 1);

- 3) a step of cooling and drying the mixture after heating; and
- 4) a step of adding an additive to the mixture after drying.

21. The method for manufacturing an oral tobacco product according to claim 20,  
wherein the following steps a) and b) are included, as a step of manufacturing the tobacco powder used for manufacturing the tobacco filler, before manufacturing the tobacco filler, and a water content of the tobacco powder is kept at 7 weight% or less after the step a) until the step b) and the step 1) set forth above:

- a) a step of drying tobacco leaves after harvesting the tobacco leaves until a water content of the tobacco leaves is 7 weight% or less; and
- b) a step of obtaining a tobacco powder by crushing the tobacco leaves after the step of drying.

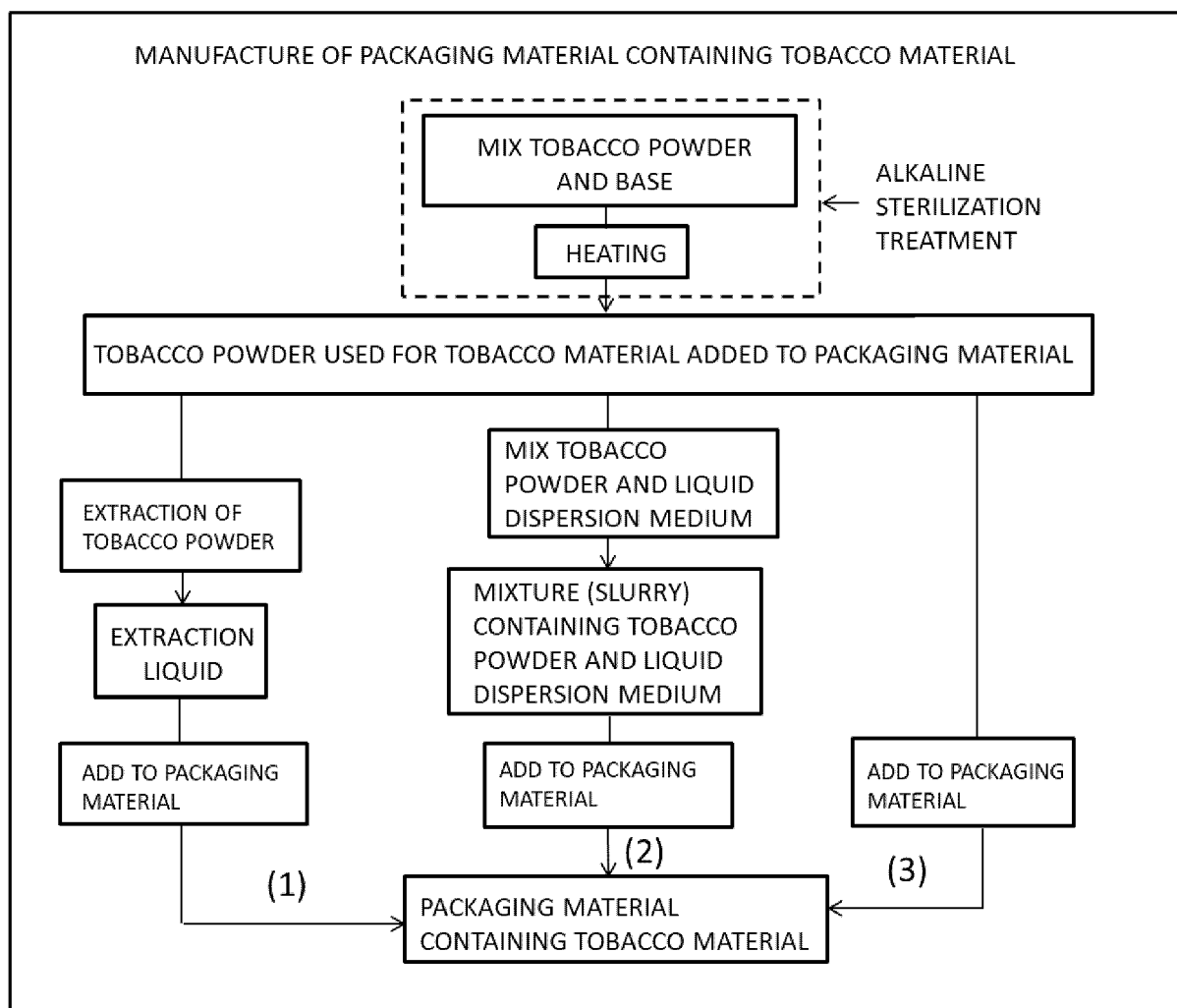


Fig. 1

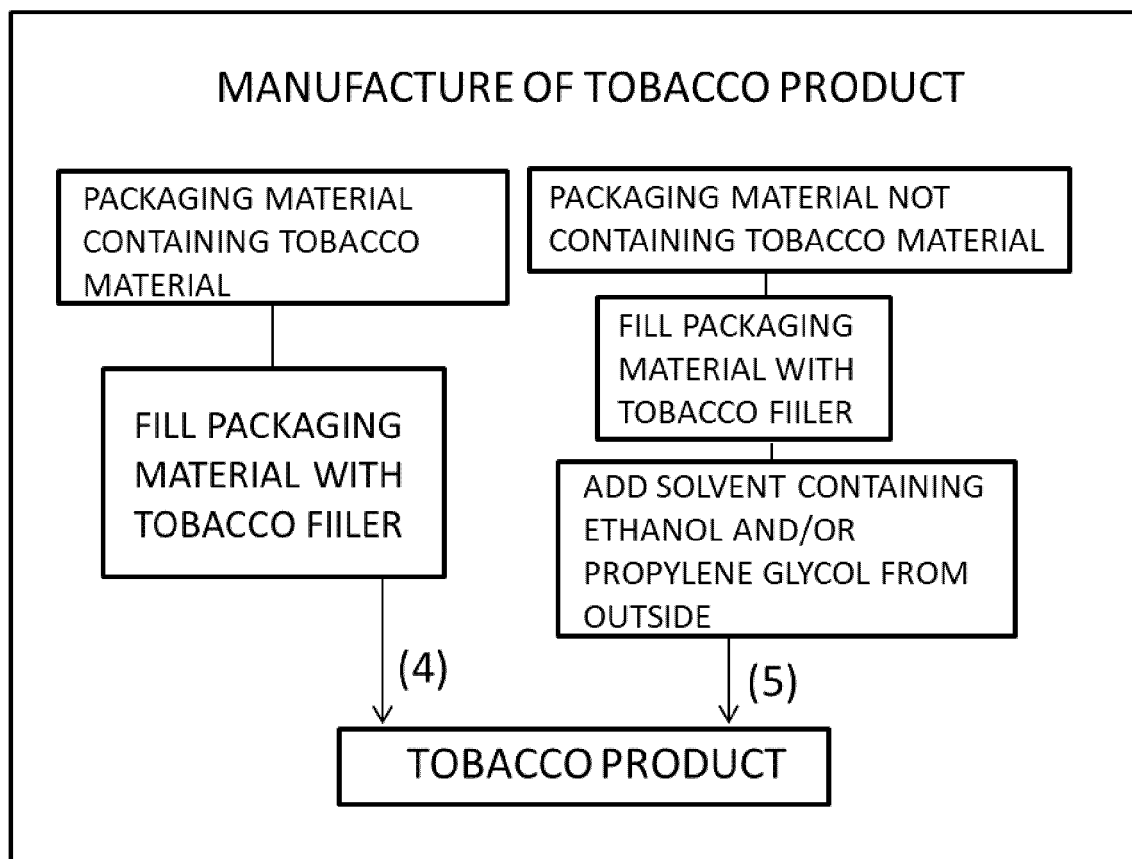


Fig. 2

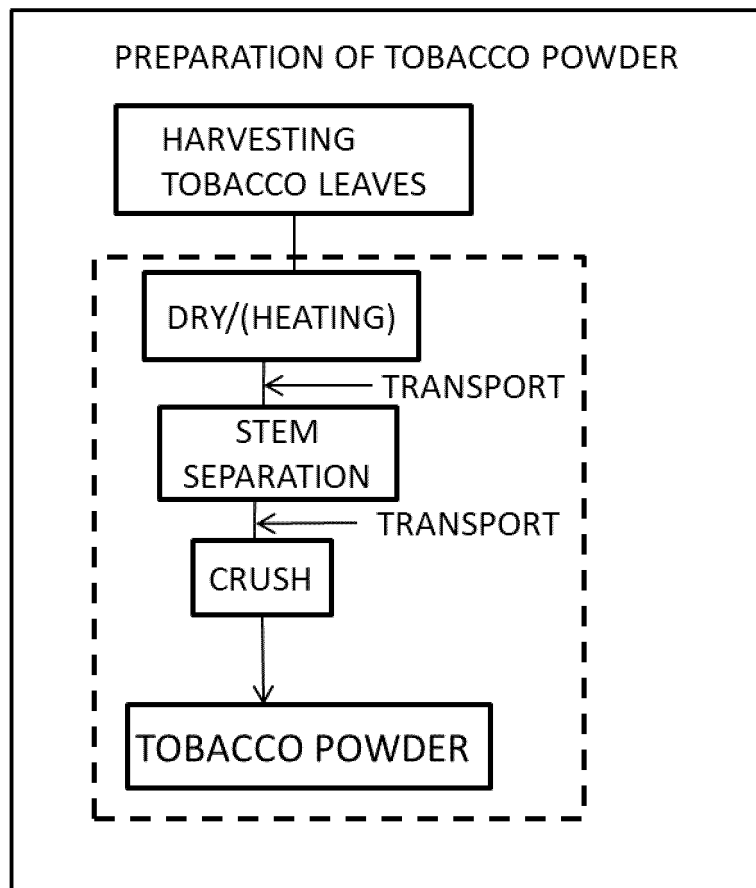


Fig. 3

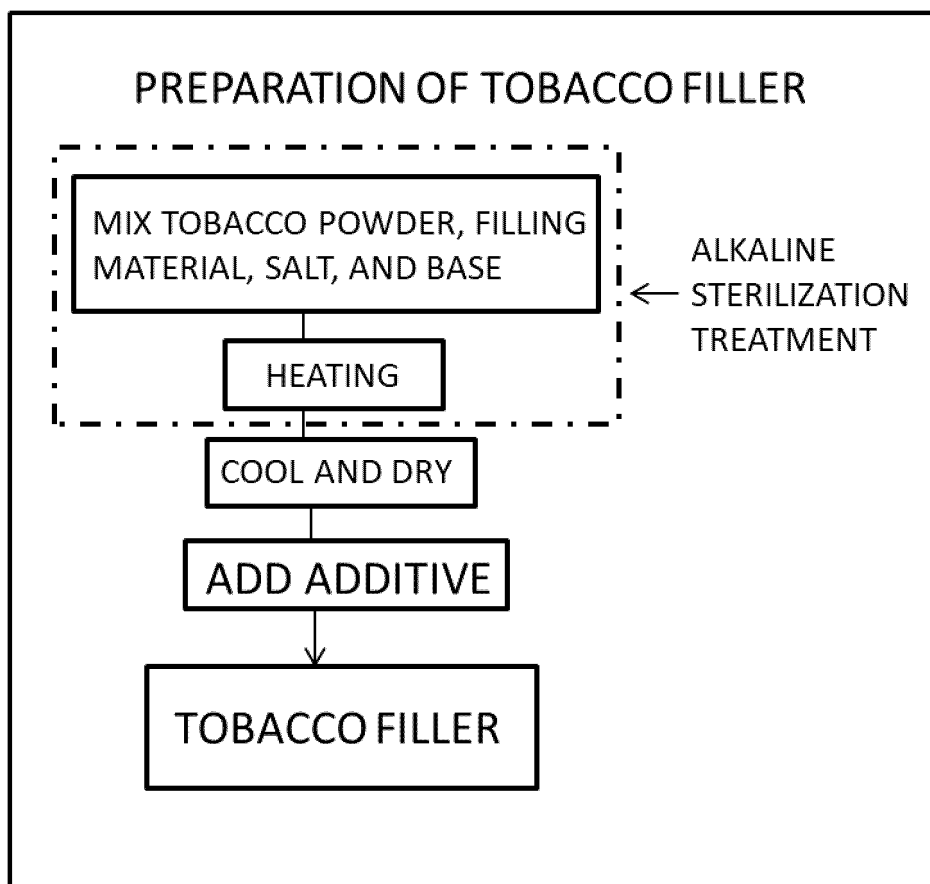


Fig. 4

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2019/008452

## A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl. A24B13/00 (2006.01) i, A24B15/26 (2006.01) i, B65D85/804 (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. A24B13/00, A24B15/26, B65D85/804

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 1971-2019

Registered utility model specifications of Japan 1996-2019

Published registered utility model applications of Japan 1994-2019

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

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2013-523102 A (PHILIP MORRIS PRODUCTS S.A.) 17 June 2013, entire text, all drawings & US 2011/0236442 A1 & WO 2011/116977 A1 & CA 2794384 A1 & KR 10-2013-0020886 A & TW 201138849 A1	1-21
A	WO 2010/114095 A1 (JAPAN TOBACCO INC.) 07 October 2010, entire text, all drawings & US 2012/00024303 A1 & EP 2415362 A1 & ES 2593112 T3 & TW 201043155 A1	1-21
A	JP 2008-538911 A (PHILIP MORRIS PRODUCTS S.A.) 13 November 2008, entire text, all drawings & US 2007/0012328 A1 & CA 2606527 A1 & KR 10-2008-0005588 A & CN 101222861 A	1-21

☐ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

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Date of the actual completion of the international search  
13.05.2019Date of mailing of the international search report  
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- JP 2010521957 W [0006]
- EP 2976951 A [0006]
- WO 2016043160 A [0120]