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(54) **METHOD FOR PRODUCING TOBACCO MATERIAL, TOBACCO MATERIAL FOR ORAL USE, AND TOBACCO PRODUCT FOR ORAL USE**

(57) There is provided a method for producing a tobacco material which comprises: a) preparing a mixed suspension of a tobacco starting material residue and an extract of a water-soluble component by immersing a tobacco starting material in water to allow the water-soluble component in the tobacco starting material to be dis-

solved in water; b) directly supplying ozone to the mixed suspension or a residue or extract obtained by subjecting the suspension to solid-liquid separation to be dissolved in it and bringing the dissolved ozone into contact with the mixed suspension.

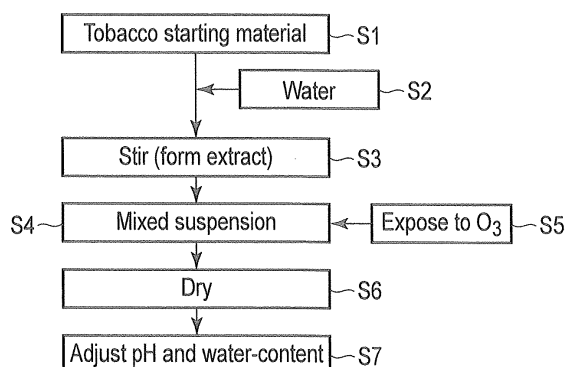


FIG. 1

Description

Technical Field

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

Background Art

10 **[0002]** Conventionally, a method comprising adding water to a tobacco starting material, heating based on tube heat, heating based on steam spraying, and drying with hot air in order to modify the tobacco material is known. In the modifying process, a saccharide or the like is added, if necessary.

15 **[0003]** However, in the method, it is not expected to modify the tobacco starting material so as to exceed the characteristics of the tobacco starting material itself. When sensory characteristics need to be largely improved, it is necessary to change the mixing ratio of the tobacco starting material and an additive.

20 **[0004]** U.S. Patent Application Publication No. 1757477 discloses a method for directly bringing a tobacco starting material into contact with an ozone gas. However, the permeability of ozone into an object to be treated is very low. Thus, even if the tobacco starting material is directly exposed to the ozone, the reaction in the tobacco starting material is not expected or a long treatment time is required. As a result, the treatment efficiency is low. This is inefficient and the treatment cost is high.

25 **[0005]** On the other hand, a tobacco product for oral use has attracted attention in recent years. The tobacco product for oral use is produced by putting a tobacco material for oral use containing moist tobacco powder in a water-permeable pouch. The tobacco product for oral use is inserted between the lip and the gum of the mouth to enjoy the taste and fragrance of the tobacco. A pH regulator is added to the moist tobacco powder so as to adjust a nearly neutral region of the pH to an alkaline region. Thus, preferable flavor and taste are provided. In the tobacco product for oral use, it is desired that the pH adjusted during the storage period is substantially maintained from the viewpoint of quality maintenance.

30 **[0006]** Jpn. PCT National Publication No. 2009-082331 discloses a tobacco product which contains magnesium carbonate as a pH regulator to make a tobacco material alkaline. The article also discloses that an additional pH regulator may be used, in addition to magnesium carbonate. As examples thereof, sodium carbonate, phosphate or the like are described. The additional pH regulator is used to rapidly adjust the pH of the tobacco material to a desired pH value (neutral or alkaline).

35 **[0007]** However, the amount of the alkaline substance to be uniquely determined is an amount required to adjust the pH of the moist tobacco powder to a desired neutral or alkaline pH value. Further, as the amount of the alkaline substance to adjust the pH of the tobacco powder to a desired pH value is relatively small, it is not possible to maintain the adjusted pH value over a long period of time. As a result, it is necessary to store the tobacco product for oral use containing the tobacco powder whose initial pH is adjusted using only the alkaline substance not at room temperature but at a low temperature (-20°C to 10°C).

40 **[0008]** If a large amount of the alkaline substance is added to the tobacco material for oral use in order to maintain the storage stability over a long period of time, the pH value increases largely. Thus, the mucous membrane of the mouth may be injured upon insertion into the mouth. The tobacco product for oral use containing the moist tobacco powder that is used in the mouth desirably has a pH of 9.0 or less.

45 **[0009]** Among tobacco products for oral use, particularly, a tobacco product for oral use in the form such that a tobacco material is wrapped with a bag-shaped pouch of non-woven fabric and the whole pouch is put into the mouth for use is known. When the pigment component as an ingredient in the tobacco product for oral use is eluted into the water content and attached to the pouch, color stains such as spots may be formed on the pouch. Further, in the pouch taken out from the mouth after use, the pigment component as an ingredient seeps into the pouch through the saliva and the pouch is colored. Such color staining of the pouch easily gives an unpleasant impression. Color stains derived from the pigment component as an ingredient impair an aesthetic appearance of the product.

50 **[0010]** A method comprising allowing only the pigment component in the tobacco starting material to be eluted to the extent that color staining of the pouch is not caused and removing it has been attempted. However, the method impairs a taste component of the tobacco starting material, and thus it has been technically difficult.

55 **[0011]** In view of such circumstances, there has been a demand for a method for removing the pigment component without impairing the flavor of tobacco.

Summary of Invention

[0012] An object of the present invention is to provide a method for producing a tobacco material in which bad habits

are removed and the bleaching or the like is modified and which has a excellent storage stability over a long period of time.

[0013] Another object of the present invention is to provide a tobacco material for oral use having a high brightness by Hunter.

[0014] Still another object of the present invention is to provide a tobacco product for oral use which contains a tobacco material for oral use having a high brightness by Hunter and exhibits an aesthetic appearance during storage and after use.

[0015] In order to solve the above problem, according to a first aspect of the present invention, there is provided a method for producing a tobacco material which comprises:

a) preparing a mixed suspension of a tobacco starting material residue and an extract of a water-soluble component by immersing a tobacco starting material in water to allow the water-soluble component in the tobacco starting material to be dissolved in water;

b) supplying ozone to the mixed suspension to be dissolved in it and bringing the dissolved ozone into contact with the mixed suspension.

[0016] According to a second aspect of the present invention, there is provided a method for producing a tobacco material which comprises:

a) immersing a tobacco starting material in water to allow the water-soluble component in the tobacco starting material to be dissolved in water;

b) separating the tobacco starting material residue after the extraction from the extract of the water-soluble component;

c) mixing the separated tobacco starting material residue with water again to prepare a suspension; and

d) supplying ozone to the suspension to be dissolved in it, and bringing the dissolved ozone into contact with the suspension.

[0017] According to a third aspect of the present invention, there is provided a method for producing a tobacco material which comprises:

a) immersing a tobacco starting material in water to allow the water-soluble component in the tobacco starting material to be dissolved in water;

b) separating the tobacco starting material residue after the extraction from the extract of the water-soluble component;

c) supplying ozone to the separated extract to be dissolved in it, and bringing the dissolved ozone into contact with the extract; and

d) drying the tobacco starting material residue and applying the extract after the ozone treatment to the tobacco starting material residue during the drying process.

[0018] According to a fourth aspect of the present invention, there is provided a tobacco material for oral use produced by the method according to any of the first to third aspects.

[0019] According to a fifth aspect of the present invention, there is provided a tobacco product for oral use which contains a tobacco material for oral use having a brightness by Hunter of 35 or more.

Brief Description of Drawings

[0020]

FIG. 1 is a flow chart showing a method for producing a tobacco material according to a first embodiment.

FIG. 2 is a flow chart showing a method for producing a tobacco material according to a second embodiment.

FIG. 3 is a flow chart showing a method for producing a tobacco material according to a third embodiment.

FIG. 4 is a flow chart showing another embodiment of the method for producing a tobacco material according to the third embodiment.

FIG. 5 is a characteristic diagram showing the storage periods and changes in pH of tobacco materials obtained in Examples 1 to 3 and Comparative Example 1.

Description of Embodiments

[0021] Hereinafter, the method for producing a tobacco material, the tobacco material for oral use, and the tobacco product for oral use according to the embodiments will be described in detail.

(First embodiment)

[0022] The method for producing a tobacco material according to the first embodiment includes the following steps.

<Step a>

[0023] A mixed suspension of a tobacco starting material residue and an extract of a water-soluble component is prepared by immersing a tobacco starting material in water to allow the water-soluble component in the tobacco starting material to be dissolved in water.

<Step b>

[0024] Ozone is supplied to the mixed suspension to be dissolved in it. The dissolved ozone is brought into contact with the mixed suspension.

[0025] According to the first embodiment, ozone is supplied to the mixed suspension of the tobacco starting material residue and the extract of the water-soluble component to be dissolved in it, and the dissolved ozone is brought into contact with the mixed suspension so that the reaction of the mixed suspension with the ozone can efficiently proceed.

[0026] When the water-soluble component in the tobacco starting material, which is eluted in water in the reaction of the mixed suspension and the ozone, is a substance which decomposes to release an acidic component (e.g., saccharide), the substance can be oxidized by efficiently bringing it into contact with the ozone. Here, the "substance which decomposes to release an acidic component" is also referred to as "a substance decomposable to release an acidic component". Further, when the substance decomposable to release an acidic component (e.g., polysaccharide) is contained in the tobacco starting material residue, the substance can be oxidized by effectively bringing it into contact with ozone. In other words, the substance decomposable to release an acidic component may be pre-oxidized before the storage of the tobacco material and released as an acidic component.

[0027] As a result, it is possible to prevent the substance decomposable to release an acidic component from being decomposed by aging and releasing the acidic component during the storage of the resulting tobacco material. This allows a decrease in the pH of the tobacco product by the acidic component to be avoided. Therefore, the pH adjusted during the storage can be substantially maintained. Thus, a tobacco material excellent in storage stability can be produced.

[0028] Further, the amount of the dissolved ozone to be brought into contact with the mixed suspension is adjusted, namely, the level of treatment for the tobacco starting material residue and the extract of the water-soluble component is selected so that it is possible to produce a tobacco material from which undesirable characteristics have been removed without impairing the flavor of tobacco, and the color tone resulting from the pigment component is adjusted (the whiteness is increased).

[0029] Particularly, a tobacco material the whiteness of which is increased, for example, to a Hunter brightness of 35 or more, may be used as a tobacco material for oral use. For the tobacco product including the tobacco material for oral use, for example, a tobacco product for oral use in the form such that a tobacco material is wrapped with a bag-shaped pouch of non-woven fabric and the whole pouch is put into the mouth for use, color staining of the pouch due to the pigment component during storage and after use is reduced. Accordingly, there can be provided a tobacco product having an improved aesthetic appearance.

[0030] Examples of the tobacco starting material used in Step a may include tobacco leaves and stems separated from the tobacco leaves. The stems are rolled with a rolling mill and finely cut. The resulting products may be used as stem shreds.

[0031] The type of water-soluble component in the tobacco starting material is not particularly limited. In general, examples thereof may include carbohydrates (saccharides), alkaloids, proteins, and amino acids.

[0032] When the tobacco starting material is immersed in water in Step a, the amount of water is preferably 5 to 20 times the amount of the tobacco starting material. Water may be at normal temperature and it is preferably heated to 20 to 60°C. Preferably, the time for immersing the tobacco starting material in water is, for example, 10 minutes to 2 hours. In immersing the tobacco starting material in water, it is preferable that both the materials are stirred to facilitate the water-soluble component in the tobacco starting material dissolving in water.

[0033] In the step of supplying ozone (Step b), the ozone is preferably dissolved in an amount of 0.5 to 500 g per 1 L of the extract in the mixed suspension. The degree of reaction of the ozone with an extract of a tobacco starting material residue and a water-soluble component, that is, the degree of treatment may be selected by changing the ozone dissolution amount.

[0034] The step of supplying ozone (Step b) is preferably performed by exposing the mixed suspension to ozone. The ozone exposure allows the tobacco starting material residue and the extract in the mixed suspension to be in contact with not only the dissolved ozone but also the bubble ozone. As a result, it is possible to effectively react the water-soluble component in the tobacco starting material residue and the extract with the ozone.

[0035] In the ozone exposure, it is desired that the ozone bubbles have a diameter of 0.5 to 1000 μm , more preferably a diameter of 0.5 to 100 μm .

[0036] In the process of bringing the dissolved ozone into contact with the mixed suspension, it is preferable that the mixed suspension is stirred, circulated with a pump or both the stirring and circulating processes are performed. The stirring and circulating processes allow the contact efficiency of the tobacco starting material residue and the extract in the mixed suspension with the ozone to be increased.

[0037] It is preferable that the step of supplying ozone is continuously performed so that the ozone dissolved in the mixed suspension as a reaction field is always present.

[0038] After Steps a and b, the mixed suspension after the ozone treatment may be dried. In this case, the mixed suspension after the ozone treatment is subjected to solid liquid separation and the tobacco starting material residue as a solid is dried. In the drying, the separated extract may be applied by spraying so that the component of the extract is incorporated into the dry product.

[0039] The pH and water content of the resulting dry product are adjusted with a pH regulator to produce a target tobacco material. As the pH regulator, for example, sodium carbonate or potassium carbonate may be used. The tobacco material after adjustment of the pH and the water content preferably has a pH of 7.0 to 9.0 and a water content of 10.0 to 50.0% by mass.

[0040] FIG. 1 shows a flow in the method for producing a tobacco material according to the first embodiment.

[0041] As shown in FIG. 1, a tobacco starting material is prepared in Step S1.

[0042] Water is added to the tobacco starting material in Step S2. The tobacco starting material is stirred in a state immersed in water at Step S3. The water-soluble component in the tobacco starting material is eluted to form an extract.

[0043] The mixed suspension of the tobacco starting material residue and the extract obtained in Step S4 is exposed to ozone (O_3) in Step S5, the ozone is dissolved, and the process of bringing the aforementioned into contact with the ozone dissolved in the mixed suspension is performed.

[0044] The mixed suspension is dried in Step S6. Thereafter, in Step S7, the pH and water content of the dry product are adjusted to produce a tobacco material.

(Second embodiment)

[0045] The method for producing a tobacco material according to the second embodiment includes the following steps.

<Step a>

[0046] A tobacco starting material is immersed in water to allow a water-soluble component in the tobacco starting material to be dissolved in water.

<Step b>

[0047] The tobacco starting material residue after the extraction is separated from the extract of the water-soluble component.

<Step c>

[0048] The separated tobacco starting material residue is again mixed with water to prepare a suspension.

<Step d>

[0049] Ozone is supplied to the suspension to be dissolved in it, and the dissolved ozone is brought into contact with the suspension.

[0050] According to the second embodiment, the water-soluble component of the tobacco starting material is extracted, the tobacco starting material residue is separated from the extract, the tobacco starting material residue is again mixed with water to prepare a suspension, ozone is supplied to the suspension to be dissolved in it, and the dissolved ozone is brought into contact with the tobacco starting material residue of the suspension so that the reaction of the tobacco starting material residue (including a large amount of the substance decomposable to release an acidic component) with the ozone can efficiently proceed.

[0051] In the reaction of the tobacco starting material residue with the ozone, the substance decomposable to release an acidic component (e.g., polysaccharide) in the tobacco starting material residue can be oxidized by effectively bringing it into contact with the ozone. In other words, the substance decomposable to release an acidic component may be pre-oxidized before the storage of the tobacco material and released as an acidic component.

[0052] As a result, it is possible to prevent the substance decomposable to release an acidic component from being decomposed by aging and releasing the acidic component during the storage of the resulting tobacco material. Thus, it is possible to avoid a decrease in the pH of the tobacco product by the acidic component. Therefore, the pH adjusted during the storage can be substantially maintained. Thus, a tobacco material excellent in storage stability can be produced.

[0053] Further, the amount of the dissolved ozone is adjusted, namely, the level of treatment for the tobacco starting material residue is selected so that it is possible to produce a tobacco material from which undesirable characteristics have been removed without impairing the flavor and the color tone resulting from the pigment component is adjusted (the whiteness is increased).

[0054] Particularly, a tobacco material the whiteness of which is increased, for example, to a Hunter brightness of 35 or more, may be used as a tobacco material for oral use. In the tobacco product including the tobacco material for oral use, for example, a tobacco product for oral use in the form such that a tobacco material is wrapped with a bag-shaped pouch of non-woven fabric and the whole pouch is put into the mouth for use, color staining of the pouch due to the pigment component during storage and after use is reduced. Accordingly, there can be provided a tobacco product having an aesthetic appearance.

[0055] Examples of the tobacco starting material used in Step a may include tobacco leaves and stems separated from the tobacco leaves. The stems are rolled with a rolling mill and finely cut. The resulting products may be used as stem shreds. However, in the second embodiment, since a target to be subjected to the ozone treatment is the tobacco starting material residue after the separation of the extract, one containing the substance decomposable to release an acidic component (e.g., polysaccharide) in the residue after the extraction of water may be selected as the tobacco starting material.

[0056] The conditions of immersing the tobacco starting material in water (in Step a) are preferably set to the same conditions as the first embodiment.

The separation step (in Step b) may be performed using, for example, a barrier filter.

[0057] In the step of supplying ozone (Step d), the ozone is preferably dissolved in an amount of 0.5 to 500 g per 1 L of the water in the mixed suspension. The degree of reaction of the ozone with a tobacco starting material residue, that is, the degree of treatment, may be selected by varying the amount of ozone dissolution.

[0058] The step of supplying ozone (Step d) is preferably performed by exposing the mixed suspension to ozone. The ozone exposure allows the tobacco starting material residue in the suspension to be in contact with not only the dissolved ozone but also the bubble ozone. Thus, it is possible to effectively react the tobacco starting material residue with the ozone.

[0059] In the ozone exposure, it is desired that the ozone bubbles have a diameter of 0.5 to 1000 μm , more preferably a diameter of 0.5 to 100 μm .

[0060] In the process of bringing the dissolved ozone into contact with the suspension, it is preferable that the suspension is stirred, circulated with a pump or both the stirring and circulating processes are performed. The stirring and circulating processes allow the contact efficiency of the tobacco starting material residue and the extract in the mixed suspension with the ozone to be increased.

[0061] It is preferable that the step of supplying ozone is continuously performed so that the ozone dissolved in the suspension as a reaction field is always present.

[0062] After Steps a and d, the suspension after the ozone treatment is dried. Preferably, the drying process is performed at 40 to 100°C so that the moisture content of the dry product is from 5.0 to 50.0% by mass.

[0063] The pH and water content of the resulting dry product are adjusted with a pH regulator to produce a target tobacco material. As the pH regulator, for example, sodium carbonate or potassium carbonate may be used. The tobacco material after adjustment of the pH and the water content preferably has a pH of 7.0 to 9.0 and a water content of 10.0 to 50.0% by mass.

[0064] In this regard, the extract separated after the extraction may not be used depending on the purpose or may be sprayed to the suspension during drying so as to return it to an original state.

[0065] FIG. 2 shows a flow in the method for producing a tobacco material according to the second embodiment.

[0066] As shown in FIG. 2, a tobacco starting material is prepared in Step S1.

[0067] Water is added to the tobacco starting material in Step S2. The tobacco starting material is stirred in a state immersed in water at Step S3. The water-soluble component in the tobacco starting material is eluted to form an extract.

[0068] The suspension after the extraction of the water-soluble component in Step S4 is subjected to solid liquid separation to obtain a tobacco starting material residue shown in Step S5 and an extract shown in Step S6.

[0069] Water is added to the tobacco starting material residue in Step S7 and a suspension is prepared in Step S8. The suspension is exposed to ozone (O_3) in Step S9, the ozone is dissolved in water, and the process of bringing the dissolved ozone into contact with the suspension is performed.

[0070] The suspension is dried in Step S10. Thereafter, the pH and water content of the dry product are adjusted in Step S11 to produce a tobacco material.

[0071] In this regard, the extract obtained in Step S6 may be returned by the spraying process during drying of the

suspension of Step S10.

(Third embodiment)

5 **[0072]** The method for producing a tobacco material according to the third embodiment includes the following steps.

<Step a>

10 **[0073]** A tobacco starting material is immersed in water to allow a water-soluble component in the tobacco starting material to be dissolved in water.

<Step b>

15 **[0074]** The tobacco starting material residue after the extraction is separated from the extract of the water-soluble component.

<Step c>

20 **[0075]** Ozone is supplied to the separated extract to be dissolved in it, and the dissolved ozone is brought into contact with the extract.

<Step d>

25 **[0076]** The tobacco starting material residue separated in Step b is dried, and the extract after the ozone treatment is applied to the tobacco starting material residue during the drying process by, for example, spraying.

[0077] According to the third embodiment, ozone is supplied to the extract of the water-soluble component extracted from the tobacco starting material to be dissolved in it, and the dissolved ozone is brought into contact with the extract so that the reaction of the extract with the ozone can efficiently proceed.

30 **[0078]** In the reaction, the substance decomposable to release an acidic component (e.g., saccharide) contained in the water-soluble component of the extract is brought into contact with the dissolved ozone so that it can be efficiently oxidized. In other words, the substance decomposable to release an acidic component may be pre-oxidized before the storage of the tobacco material and released as an acidic component.

35 **[0079]** As a result, during drying of the separated tobacco starting material residue, the extract after the ozone treatment (including the substance decomposable to release an acidic component which has been already oxidized) is applied so as to return it to the residue. Thus, it is possible to prevent the substance decomposable to release an acidic component from being decomposed by aging and releasing the acidic component during the storage of the resulting tobacco material. Thus, it is possible to avoid a decrease in the pH of the tobacco product by the acidic component. Therefore, the pH adjusted during the storage can be substantially maintained. Thus, a tobacco material excellent in storage stability can be produced.

40 **[0080]** Further, the amount of the dissolved ozone is adjusted, namely, the level of treatment for the extract of the water-soluble component is selected so that undesirable characteristics can be removed without impairing the flavor and the color tone resulting from the pigment component of the extract can be adjusted (the whiteness is increased). As a result, during drying of the tobacco starting material residue after the separation, the extract after the ozone treatment is returned to the residue by the spraying process so that it is possible to produce a tobacco material in which the flavor is maintained, there are no undesirable characteristics, and the whiteness is increased.

45 **[0081]** Particularly, a tobacco material of which the whiteness is increased, to for example, a Hunter brightness of 35 or more may be used as a tobacco material for oral use. In the tobacco product including the tobacco material for oral use, for example, a tobacco product for oral use in the form such that a tobacco material is wrapped with a bag-shaped pouch of non-woven fabric and the whole pouch is put into the mouth for use, color staining of the pouch due to the pigment component during storage and after use is reduced. Accordingly, there can be provided a tobacco product having a good aesthetic appearance.

50 **[0082]** Examples of the tobacco starting material used in Step a may include tobacco leaves and stems separated from the tobacco leaves. The stems are rolled with a rolling mill and finely cut. The resulting products may be used as stem shreds. However, in the third embodiment, since a target to be subjected to the ozone treatment is the extract, one containing the substance decomposable to release an acidic component (e.g., saccharide) in the extract may be selected as the tobacco starting material to be subjected to the treatment.

[0083] The conditions in immersing the tobacco starting material in water (in Step a) are preferably set to the same conditions as the first embodiment.

[0084] The separation step (in Step b) may be performed using, for example, a barrier filter.

[0085] In the step of supplying ozone (Step c), the ozone is preferably dissolved in an amount of 0.5 to 500 g per 1 L of the extract. The degree of reaction of the ozone with a starting material, that is, the degree of treatment may be selected by varying the amount of ozone dissolution.

[0086] The step of supplying ozone (Step c) is preferably performed by exposing the extract to ozone. The ozone exposure allows the extract to be in contact with not only the dissolved ozone but also the bubble ozone. Thus, it is possible to effectively react the extract with the ozone. In the ozone exposure, it is desired that the ozone bubbles have a diameter of 0.5 to 1000 μm , more preferably a diameter of 0.5 to 100 μm .

[0087] In the process of bringing the dissolved ozone into contact with the extract, it is preferable that the extract is stirred, circulated with a pump or both the stirring and circulating processes are performed. The stirring and circulating processes allow the contact efficiency of the extract with the ozone to be increased.

[0088] It is preferable that the step of supplying ozone is continuously performed so that the ozone dissolved in the extract as a reaction field is always present.

[0089] The process of drying the separated tobacco starting material residue in Step d is preferably performed at 40 to 100°C because the extract treated with the ozone during the drying process is returned by the spraying process. Preferably, the drying process is performed so that the moisture content of the resulting dry product is from 5.0 to 25.0% by mass.

[0090] In Step d, a partial or total amount of the extract (the extract treated with the ozone) may be applied to the tobacco starting material residue during the drying process.

[0091] The pH and water content of the dry product obtained in Steps a to d are adjusted with a pH regulator to produce a target tobacco material. As the pH regulator, for example, sodium carbonate or potassium carbonate may be used. The tobacco material after adjustment of the pH and the water content preferably has a pH of 7.0 to 9.0 and a water content of 10.0 to 50.0% by mass.

[0092] FIG. 3 shows a flow in the method for producing a tobacco material according to the third embodiment.

[0093] As shown in FIG. 3, a tobacco starting material is prepared in Step S1.

[0094] Water is added to the tobacco starting material in Step S2. The tobacco starting material is stirred in a state immersed in water at Step S3. The water-soluble component in the tobacco starting material is eluted to form an extract.

[0095] The suspension after the extraction of the water-soluble component in Step S4 is subjected to solid liquid separation to obtain a tobacco starting material residue shown in Step S5 and an extract shown in Step S6.

[0096] The extract is exposed to ozone (O_3) in Step S7, the ozone is dissolved in water, and the process of bringing the dissolved ozone into contact with the extract is performed.

[0097] The tobacco starting material residue after solid liquid separation in Step S8 is dried, and the extract after the ozone treatment is returned to the tobacco starting material residue during the drying process by the spraying process. Thereafter, in Step S9, the pH and water content of the dry product are adjusted to produce a tobacco material.

[0098] In the third embodiment, the following steps may be added.

[0099] The separated tobacco starting material residue is again mixed with water to prepare a suspension. Ozone is supplied to the suspension to be dissolved in it, and the dissolved ozone is brought into contact with the suspension. The suspension after the ozone treatment is dried, and the extract after the ozone treatment is, for example, returned to the tobacco starting material residue dry product during drying, by, for example, the spraying process.

[0100] FIG. 4 shows a flow in the method for producing a tobacco starting material.

[0101] As shown in FIG. 4, a tobacco starting material is prepared in Step S1.

[0102] Water is added to the tobacco starting material in Step S2. The tobacco starting material is stirred in a state immersed in water at Step S3. The water-soluble component in the tobacco starting material is eluted to form an extract.

[0103] The suspension after the extraction of the water-soluble component in Step S4 is subjected to solid liquid separation to obtain a tobacco starting material residue shown in Step S5 and an extract shown in Step S6.

[0104] Water is added to the tobacco starting material residue in Step S7 and a suspension is prepared in Step S8. The suspension is exposed to ozone (O_3) in Step S9, the ozone is dissolved in water, and the process of bringing the dissolved ozone into contact with the suspension is performed.

[0105] The suspension is dried in Step S10.

[0106] The extract obtained by solid liquid separation in Step S4 is exposed to ozone (O_3) in Step S11, the ozone is dissolved in water, and the process of bringing the dissolved ozone into contact with the extract is performed.

[0107] During drying of the suspension in Step S10, the extract after the ozone treatment is returned by the spraying process. Thereafter, the pH and water content of the dry product are adjusted in Step S12 to produce a tobacco material.

[0108] According to another embodiment of the third embodiment, appropriate ozone treatment may be performed depending on the properties of the tobacco starting material residue after the extraction and the properties of the extract.

[0109] That is, in the first embodiment, the ozone treatment is performed on the mixed suspension of the tobacco starting material residue and the extract after the extraction. The ozone effect is uniformly applied to the tobacco starting material residue and the extract.

[0110] However, a large amount of the substance decomposable to release an acidic component may be contained in the tobacco starting material residue after the extraction, depending on the kind of the tobacco starting material. In contrast, a large amount of the substance decomposable to release an acidic component may be contained in the extract. Further, the flavor, undesirable characteristics, and the pigment component may be weighted in either the tobacco

starting material residue or the extract.
[0111] As described above, the tobacco starting material residue and the extract are respectively treated with the ozone so that the degree of the reaction of the tobacco starting material residue and the extract with the ozone, namely, the treatment degree can be independently and arbitrarily adjusted according to their properties. Thus, it is possible to produce a tobacco material of which the storage stability is excellent, the flavor is maintained, there are no undesirable characteristics, and the whiteness is increased.

(Fourth embodiment)

[0112] The tobacco product for oral use according to the fourth embodiment includes a tobacco material for oral use having a Hunter brightness of 35 or more. A more preferred tobacco material for oral use has a Hunter brightness of 37 or more.

[0113] The tobacco product for oral use has, for example, a structure in which a tobacco material is wrapped with a bag-shaped pouch of non-woven fabric. As for the tobacco product for oral use, the whole pouch is put into the mouth for use.

[0114] The tobacco material for oral use may be obtained by the method for producing a tobacco material described in the first to third embodiments.

[0115] Also, the tobacco material for oral use may be obtained by treating the tobacco starting material with an oxidant except for the ozone. As the oxidant, for example, hydrogen peroxide may be used.

[0116] Since the tobacco product for oral use according to the fourth embodiment includes a tobacco material for oral use having a Hunter brightness of 35 or more, color staining of the pouch due to the pigment component during storage and after use is reduced and an aesthetic appearance is exhibited.

[0117] Hereinafter, examples and comparative examples of the present invention will be described. In this regard, the moisture content of the tobacco materials according to Comparative Examples 1 and 2 and Examples 1 to 4 was measured by the following method.

<Measurement method of moisture content>

[0118] The moisture content of 3.0 g of a powdered tobacco material was measured using a heat drying type moisture meter (HB 43-S: manufactured by METTLER TOLEDO International Inc.).

(Comparative Example 1)

[0119] First, a tobacco starting material in which ground powders of rustica leaves, burley leaves, and tobacco stems were mixed at a proportion of 35% by mass, 15% by mass, and 50% by mass, respectively, was prepared.

[0120] Subsequently, 4 kg of the tobacco starting material was heated at 110°C for 30 minutes, and heated and dried at 80°C for 3 hours. Further, the dry product was cooled. The moisture content of the resulting tobacco starting material was then measured. As a result, the moisture content of the tobacco starting material was 18% by mass.

[0121] Subsequently, water (in an amount required to adjust the final moisture content of the tobacco material to 25.0% by mass) was prepared. Sodium carbonate was added to the water in an amount of 3.0% by mass based on the dry weight of the tobacco material. The total amount of the resulting sodium carbonate solution was sprayed to the tobacco starting material to produce a tobacco material having a moisture content of 25% by mass and a pH (initial value) of 8.43.

(Example 1)

[0122] Four kg of a tobacco starting material with the same composition as that of Comparative Example 1 and 80 L of water were stirred and mixed for 30 minutes so as to allow the component in the tobacco starting material to be eluted (dissolved) in water.

[0123] Subsequently, the stirring was continued and the exposure to ozone fine bubbles was continued for 3 hours while circulating the mixed suspension of the tobacco starting material residue and the extract with a pump. At this time, the average diameter of the ozone fine bubbles was 10 μm and the exposure to ozone was carried out at 25°C and 38.0 g/hr.

[0124] Subsequently, the mixed suspension after the ozone treatment was subjected to solid-liquid separation. In the drying process, the extract as a liquid was sprayed to the tobacco starting material residue while heating the tobacco

starting material residue as a solid to 80°C. The moisture content of the resulting dry product was measured. As a result, the moisture content of the dry product was 8.8% by mass.

[0125] Subsequently, water (in an amount required to adjust the final moisture content of the tobacco material to 25.0% by mass) was prepared. Sodium carbonate was added to the water in an amount of 2.3% by mass based on the dry weight of the tobacco material. The total amount of the resulting sodium carbonate solution was sprayed to the dry product to produce a tobacco material having a moisture content of 25% by mass and a pH (initial value) of 8.50.

(Example 2)

[0126] First, a tobacco starting material in which ground powders of rustica leaves burley leaves were mixed at a proportion of 70% by mass and 30% by mass, respectively, was prepared.

[0127] Then, 4 kg of a tobacco starting material and 80 L of water were stirred and mixed for 30 minutes so as to allow the component in the tobacco starting material to be eluted (dissolved) in water. Subsequently, the stirring was continued and the exposure to ozone fine bubbles was continued for 3 hours while circulating the mixed suspension of the tobacco starting material residue and the extract with a pump. At this time, the average diameter of the ozone fine bubbles was 10 µm and the exposure to ozone was carried out at 25°C and 38.0 g/hr.

[0128] Subsequently, the mixed suspension after the ozone treatment was subjected to solid-liquid separation. In the drying process, the extract as a liquid was sprayed to the tobacco starting material residue while heating the tobacco starting material residue as a solid to 80°C. The moisture content of the resulting dry product was measured. As a result, the moisture content of the dry product was 8.5% by mass.

[0129] Subsequently, water (in an amount required to adjust the final moisture content of the tobacco material to 25.0% by mass) was prepared. Sodium carbonate was added to the water in an amount of 2.8% by mass based on the dry weight of the tobacco material. The total amount of the resulting sodium carbonate solution was sprayed to the dry product to produce a tobacco material having a moisture content of 25% by mass and a pH (initial value) of 8.25.

(Example 3)

[0130] Four kg of a tobacco starting material with the same composition as that of Example 2 and 80 L of water were stirred and mixed for 30 minutes so as to allow the component in the tobacco starting material to be eluted (dissolved) in water. Subsequently, the stirring was continued and the exposure to ozone fine bubbles was continued for 3 hours while circulating the mixed suspension of the tobacco starting material residue and the extract with a pump. At this time, the average diameter of the ozone fine bubbles was 10 µm and the exposure to ozone was carried out at 25°C and 38.0 g/hr.

[0131] Subsequently, the mixed suspension after the ozone treatment was subjected to solid-liquid separation. In the drying process, the extract as a liquid was sprayed to the tobacco starting material residue while heating the tobacco starting material residue as a solid to 80°C. The moisture content of the resulting dry product was measured. As a result, the moisture content of the dry product was 8.5% by mass.

[0132] Subsequently, water (in an amount required to adjust the final moisture content of the tobacco material to 50.0% by mass) was prepared. Sodium carbonate was added to the water in an amount of 2.8% by mass based on the dry weight of the tobacco material. The total amount of the resulting sodium carbonate solution was sprayed to the dry product to produce a tobacco material having a moisture content of 25% by mass and a pH (initial value) of 8.48.

[0133] The smell of the resulting tobacco materials of Comparative Example 1 and Example 1 was measured by the following method. The results are shown in Table 1 below.

<Method for measuring smell>

1) Analytical laboratories: Japan Food Research Laboratories

2) Preparation of sample gas

[0134] Ten g of a sample was placed in a sample bag of polyethylene terephthalate (volume: about 2 L), and the inside of the bag was substituted with a nitrogen gas. Thereafter, it was allowed to stand at room temperature for about 3 hours and used as a sample gas.

3) Measurement method

[0135] The smell of the sample gas was measured under the following conditions using a smell identification testing device.

4) Measurement conditions

[0136]

Model: Smell identification device: FF-2A (Shimadzu Corporation.)
 Autosampler for sample bags: FAS-1 (Shimadzu Corporation.)
 Dilution mixing device: FDL-1 (Shimadzu Corporation.)
 Sampling tube: usually used
 Software for data analyses: Smell P (Shimadzu Corporation.)

[Table 1]

System and kind of smell (Standard gas component)	Odor contribution	
	Comparative Example 1	Example 1
Ammonia	2.3	-5.3
Note) the odor contribution is a value obtained by representing the strength of the smell of the system as an odor index equivalent value.		

[0137] When the calculated value is the set threshold or less, it is represented by a minus (-) value (the lowest value: -30).

[0138] As is clear from Table 1 above, undesirable characteristics in the tobacco material obtained in Example 1 can be significantly reduced as compared to those in the tobacco material of Comparative Example 1.

[0139] In this regard, undesirable characteristics in the tobacco materials obtained in Examples 2 and 3 were significantly reduced, similarly to Example 1.

[0140] Regarding the tobacco materials obtained in Examples 1 to 3 and Comparative Example 1, the pH during the production, the pH after storage for two months, and the pH after storage for four months were measured by the following method. The results are shown in FIG. 5. In the storage process, the tobacco material was placed in an atmosphere where the temperature went up and down every day (25 to 30°C).

<pH measurement method>

[0141] Three g of a tobacco material was weighed and put into a vial, and 30 mL of water was added thereto. The mixture was subjected to shaking at 200 rpm for 10 minutes (extraction treatment). The resulting mixture was allowed to stand for 5 minutes, and then the pH of the extract was measured using a pH meter (IQ 240, manufactured by IQ Scientific Instruments Inc.).

[0142] As is clear from FIG. 5, the pH during storage in the tobacco materials obtained in Examples 1 to 3 can be significantly controlled as compared to that of the tobacco material obtained in Comparative Example 1. In other words, changes in the pH during storage are small.

(Examples 4 to 9)

[0143] Five kg of a tobacco starting material with the same composition as that of Comparative Example 1 and 400 mL of water were stirred and mixed for 30 minutes so as to allow the component in the tobacco starting material to be eluted (dissolved) in water.

[0144] Subsequently, the stirring was continued and the exposure to ozone fine bubbles was continued for 10 minutes, for 20 minutes, for 30 minutes, for 40 minutes, for 80 minutes, and 120 minutes while circulating the mixed suspension of the tobacco starting material residue and the extract with a pump. At this time, the average diameter of the ozone fine bubbles was 45 μm and the exposure to ozone was carried out at 25°C and 6.0 g/hr.

[0145] Subsequently, the mixed suspension after the ozone treatment was subjected to solid-liquid separation. The extract after the separation was concentrated. The concentrated extract was sprayed to the tobacco starting material residue after the separation so as to make it return to the original state while keeping the residue at 40°C, and thus six kinds of tobacco materials were produced.

(Comparative Example 2)

[0146] A tobacco material was produced in the same manner as Examples 4 to 9 except that the circulation of the

mixed suspension of the tobacco starting material residue and the extract with a pump and the exposure to ozone fine bubbles were not performed.

[0147] Regarding the resulting tobacco materials of Examples 4 to 9 and Comparative Example 2, the Hunter brightness was measured by the following method. The results are shown in Table 2 below.

<Method for measuring Hunter brightness>

[0148] A powdered tobacco material was collected and placed in a petri dish having a diameter of 12 cm until the thickness was about 2 cm. The brightness, hue, and saturation were measured with a hue color difference meter (Hue color difference meter CR410, manufactured by Konica Minolta, Inc.).

[0149] Based on the resulting data, the Hunter brightness was calculated using the following equation.

$$\text{Hunter brightness} = 100 - \sqrt{(100 - L)^2 + (a^2 + b^2)}$$

[0150] Here, L represents brightness, and a and b represent chromaticity showing hue and saturation.

[Table 2]

	Circulation time of mixed suspension (time for exposure to ozone) (min.)	Hunter brightness
Comparative Example 2	0	34.7
Example 4	10	37
Example 5	20	40.0
Example 6	30	40.7
Example 7	40	42.5
Example 8	80	45.1
Example 9	120	45.4

[0151] As is clear from Examples 4 to 9 of Table 2 above, the longer the circulation time of the mixed suspension (the time for exposure to ozone), the higher Hunter brightness of the resulting tobacco material is.

(Examples 10 to 12)

[0152] Five g of a tobacco starting material with the same composition as that of Comparative Example 1 and 400 mL of water were stirred and mixed for 30 minutes so as to allow the component in the tobacco starting material to be eluted (dissolved) in water.

[0153] Subsequently, while the stirring was continued, the temperature of the mixed suspension of the tobacco starting material residue and the extract was set to 5°C, 25°C, and 80°C. The exposure to ozone fine bubbles was continued for 5 hours while circulating the mixed suspension with a pump. At this time, the average diameter of the ozone fine bubbles was 45 μm and the exposure to ozone was carried out at 6.0 g/hr.

[0154] Subsequently, the mixed suspension after the ozone treatment was subjected to solid-liquid separation. The extract after the separation was concentrated. The concentrated extract was sprayed to the tobacco starting material residue after the separation so as to make it return to the original state while keeping the residue at 40°C, and thus three kinds of tobacco materials were produced.

[0155] Regarding the resulting tobacco materials of Examples 10 to 12, the pH and the Hunter brightness were measured by the above method. The results are shown in Table 3 below.

[Table 3]

	Temperature of circulation treatment of mixed suspension (°C)	pH of tobacco material	Hunter brightness
Example 10	5	4.7	51.3
Example 11	25	5	51.8
Example 12	80	5.2	46.3

[0156] As is clear from Table 3 above, the tobacco materials of Examples 9 and 10 in which the temperature of the mixed suspension was set to 5°C and 25°C showed a high Hunter brightness as compared to that of the tobacco material of Example 11 in which the temperature of the mixed suspension was set to 80°C. This is because the solubility of ozone is higher at the low temperature side.

(Comparative Example 3)

[0157] A tobacco starting material in which ground powders of rustica leaves, burley leaves, and tobacco stems were mixed at a proportion of 35% by mass, 15% by mass, and 50% by mass, respectively, was prepared.

[0158] Then, 4 kg of a tobacco starting material and 80 L of water were stirred and mixed for 30 minutes so as to allow the component in the tobacco starting material to be eluted (dissolved) in water. Subsequently, the suspension was subjected to solid-liquid separation and only the tobacco starting material residue as a solid was heated and dried at 80°C. The moisture content of the dry product was measured. As a result, the moisture content of the dry product was 9.8% by mass.

[0159] Subsequently, water (in an amount required to adjust the final moisture content of the tobacco material to 25.0% by mass) was prepared. The water was sprayed to the dry product to produce a tobacco material having a moisture content of 25.2% by mass.

(Example 13)

[0160] Four kg of a tobacco starting material with the same composition as that of Comparative Example 3 and 80 L of water were stirred and mixed for 30 minutes so as to allow the component in the tobacco starting material to be eluted (dissolved) in water. Subsequently, the suspension was separated and only the tobacco starting material residue as a solid was collected. Eighty L of water was added to the tobacco starting material residue again and the stirring was continued. The exposure to ozone fine bubbles was continued for 3 hours while circulating the suspension of the tobacco starting material residue and the water with a pump. At this time, the average diameter of the ozone fine bubbles was 10 μm and the exposure to ozone was carried out at 25°C and 38.0 g/hr.

[0161] Subsequently, the suspension after the ozone treatment was subjected to solid-liquid separation and only the tobacco starting material residue as a solid was collected. The residue was heated and dried at 80°C. The moisture content of the dry product was measured. As a result, the moisture content of the dry product was 9.4% by mass.

[0162] Subsequently, water (in an amount required to adjust the final moisture content of the tobacco material to 25.0% by mass) was prepared. The water was sprayed to the dry product to produce a tobacco material having a moisture content of 24.7% by mass.

[0163] Regarding the resulting tobacco materials of Comparative Example 3 and Example 13, the Hunter brightness was measured by the above method. The results are shown in Table 4 below.

[Table 4]

	Hunter brightness
Comparative Example 3	35
Example 13	47

[0164] As is clear from Table 4 above, the tobacco material obtained in Example 13 shows a high Hunter brightness as compared to that of the tobacco material obtained in Comparative Example 3.

(Example 14)

[0165] First, a tobacco starting material consisting of 60% by mass of laminae (18% by mass of burley and 42% by mass of rustica) and 40% by mass of stems was prepared.

[0166] 5 g of the tobacco starting material and 400 mL of water were stirred and mixed for 30 minutes so as to allow the component in the tobacco starting material to be eluted or dissolved in water. Subsequently, the mixed suspension was filtered to be separated into an extract and a tobacco starting material residue.

[0167] The exposure to ozone fine bubbles was continued for 120 minutes while circulating the extract with a pump. At this time, the average diameter of the ozone fine bubbles was 45 μm and the exposure to ozone was carried out at 25°C and 6.0 g/hr. After the treatment, the ozone was concentrated to a volume of 10% or less under reduced pressure at 60°C.

[0168] Subsequently, the total amount of the extract after the ozone treatment was sprayed for drying while heating

the tobacco starting material residue at 40°C (RH 60%). The resulting product was allowed to stand at 22°C and 60% RH for 12 hours to produce a tobacco material having a moisture content of 14.6% by mass and a pH of 3.726.

(Example 15)

[0169] 5 g of the tobacco starting material having the same composition as that of Example 14 and 400 mL of water were stirred and mixed for 30 minutes so as to allow the component in the tobacco starting material to be eluted or dissolved in water. Subsequently, the mixed suspension was filtered to be separated into an extract and a tobacco starting material residue.

[0170] The exposure to ozone fine bubbles was continued for 120 minutes while circulating the extract with a pump. At this time, the average diameter of the ozone fine bubbles was 45 μm and the exposure to ozone was carried out at 25°C and 6.0 g/hr. After the treatment, the ozone was concentrated to a volume of 10% or less under reduced pressure at 60°C.

[0171] On the other hand, 400 mL of water was newly added to the tobacco starting material residue to prepare a suspension. The exposure to ozone fine bubbles was continued for 120 minutes while circulating the suspension with a pump. At this time, the average diameter of the ozone fine bubbles was 45 μm and the exposure to ozone was carried out at 25°C and 6.0 g/hr. Thereafter, it was dried to obtain a treated residue.

[0172] Subsequently, the total amount of the extract after the ozone treatment was sprayed for drying while heating the treated residue at 40°C (RH 60%). The resulting product was allowed to stand at 22°C and 60% RH for 12 hours to produce a tobacco material having a moisture content of 12.2% by mass and a pH of 3.227.

[0173] Regarding the resulting tobacco materials of Examples 14 and 15, the Hunter brightness was measured by the above method. The results are shown in Table 5 below.

[Table 5]

	Hunter brightness
Example 14	41.5
Example 15	47.7

[0174] As is clear from Table 5 above, the tobacco material of Example 14 obtained by spraying the extract after the ozone treatment to the tobacco starting material residue after the extraction exhibits a high Hunter brightness. Further, the tobacco material of Example 15 obtained by spraying the extract after the ozone treatment to the tobacco starting material residue treated with the ozone after the extraction and drying it exhibits a high Hunter brightness as compared to that of the tobacco material of Example 14.

(Example 16)

[0175] One g of the tobacco starting material having the same composition as that of Example 14 was mixed with 5% of hydrogen peroxide water to prepare a mixed suspension. The suspension was allowed to stand for 12 hours, followed by filtration of the mixed suspension to obtain a residue. Subsequently, the treated residue was dried while heating the residue at 40°C (RH 60%). Thereafter, the resulting product was allowed to stand at 22°C and 60% RH for 12 hours to produce a tobacco material. The Hunter brightness of the resulting tobacco material was measured, and it was 47.1.

[0176] As is clear from this result, even if hydrogen peroxide is used as an oxidant except for the ozone, a high Hunter brightness can be similarly obtained.

Industrial Applicability

[0177] According to the present invention, there can be provided a method for producing a tobacco material in which undesirable characteristics are removed and the bleaching or the like is modified and which has a excellent storage stability over a long period of time as well as a tobacco material for oral use having a high Hunter brightness.

[0178] According to the present invention, there can be provided a tobacco product for oral use which contains a tobacco material for oral use having a high Hunter brightness, and exhibits an aesthetic appearance during storage and after use.

Claims

1. A method for producing a tobacco material comprising:

- 5 a) preparing a mixed suspension of a tobacco starting material residue and an extract of a water-soluble component by immersing a tobacco starting material in water to allow the water-soluble component in the tobacco starting material to be dissolved in water;
 b) supplying ozone to the mixed suspension to be dissolved in it and bringing the dissolved ozone into contact with the mixed suspension.

10 2. The method for producing a tobacco material according to claim 1, wherein the b) comprises bringing the dissolved ozone into contact with the mixed suspension while circulating the mixed suspension.

15 3. A method for producing a tobacco material comprising:

- a) immersing a tobacco starting material in water to allow the water-soluble component in the tobacco starting material to be dissolved in water;
 b) separating the tobacco starting material residue after the extraction from the extract of the water-soluble component;
 20 c) mixing the separated tobacco starting material residue with water again to prepare a suspension; and
 d) supplying ozone to the suspension to be dissolved in it, and bringing the dissolved ozone into contact with the suspension.

25 4. The method for producing a tobacco material according to claim 3, wherein the d) comprises bringing the dissolved ozone into contact with the suspension while circulating the suspension.

5. A method for producing a tobacco material comprising:

- 30 a) immersing a tobacco starting material in water to allow the water-soluble component in the tobacco starting material to be dissolved in water;
 b) separating the tobacco starting material residue after the extraction from the extract of the water-soluble component;
 c) supplying ozone to the separated extract to be dissolved in it, and bringing the dissolved ozone into contact with the extract; and
 35 d) drying the tobacco starting material residue and returning the extract after the ozone treatment to the tobacco starting material residue during the drying process.

6. The method for producing a tobacco material according to claim 5, further comprising:

- 40 mixing the separated tobacco starting material residue with water again to prepare a suspension;
 supplying ozone to the suspension to be dissolved in the suspension, and bringing the dissolved ozone into contact with the suspension; and
 heating and drying the suspension after the ozone treatment and returning the extract after the ozone treatment to the tobacco starting material residue during the drying process.

45 7. The method for producing a tobacco material according to any one of claims 1 to 6, wherein the supplying ozone is carried out by exposure to ozone.

50 8. The method for producing a tobacco material according to claim 7, wherein the ozone bubbles formed by the exposure to ozone have a diameter of 0.5 to 1000 μm .

9. The method for producing a tobacco material according to any one of claims 1 to 8, wherein the ozone supplying is continuously performed so that the ozone dissolved in a reaction field is always present.

55 10. A tobacco material for oral use which is produced by the method according to any one of claims 1 to 9.

11. A tobacco product for oral use comprising a tobacco material for oral use which has a Hunter brightness by of 35 or more.

12. The tobacco product for oral use according to claim 11, wherein the tobacco material for oral use is treated with an oxidant.

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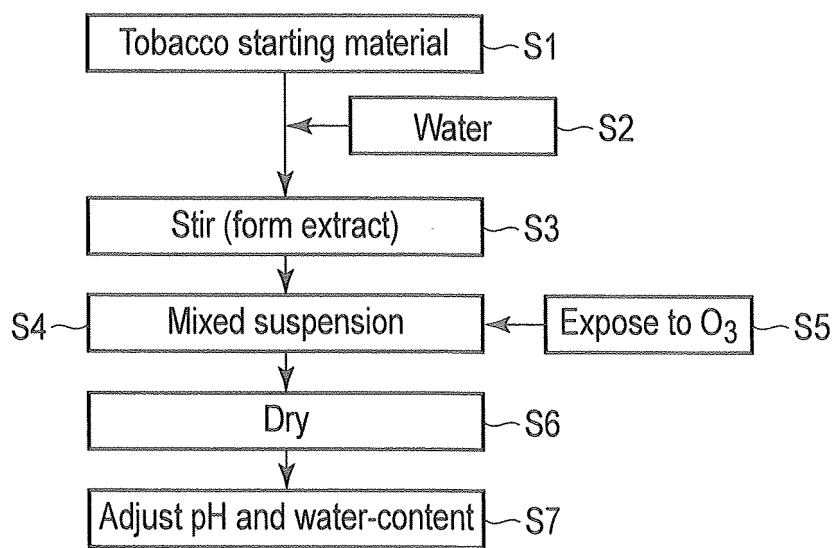


FIG. 1

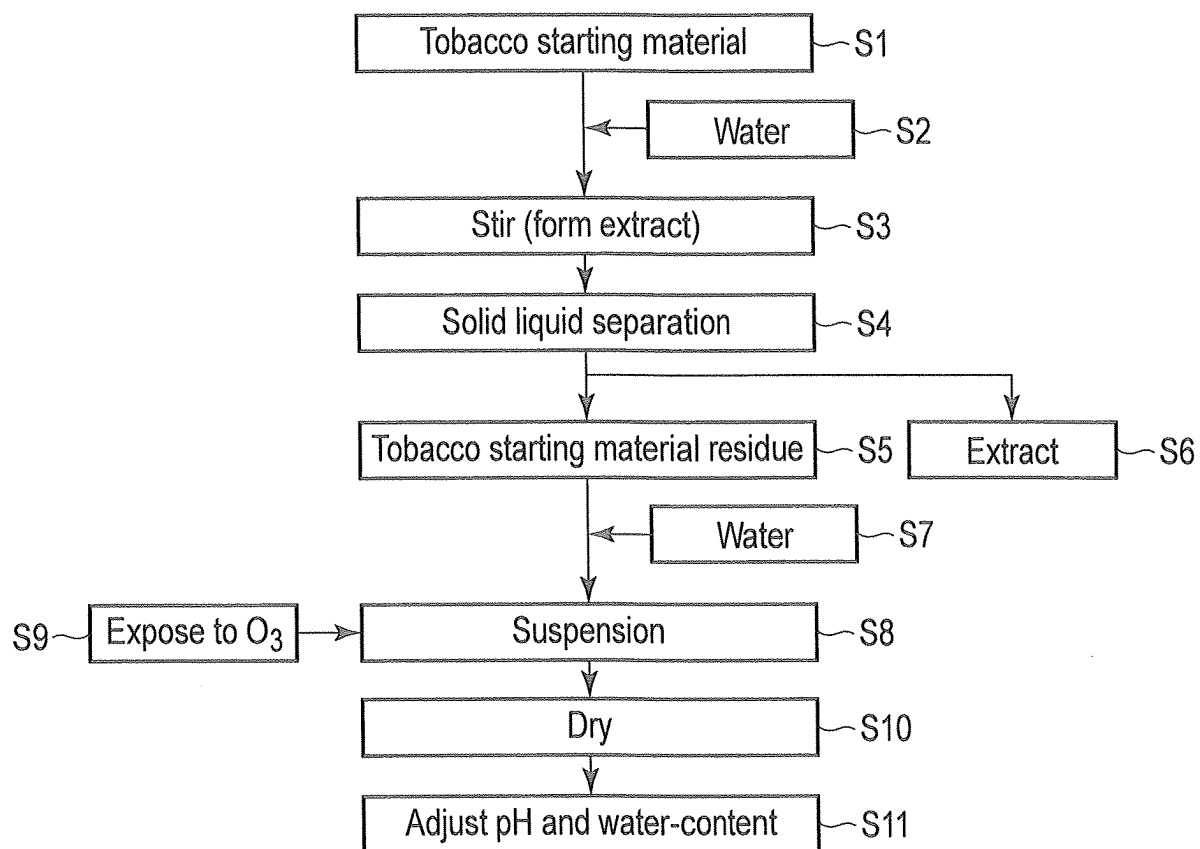


FIG. 2

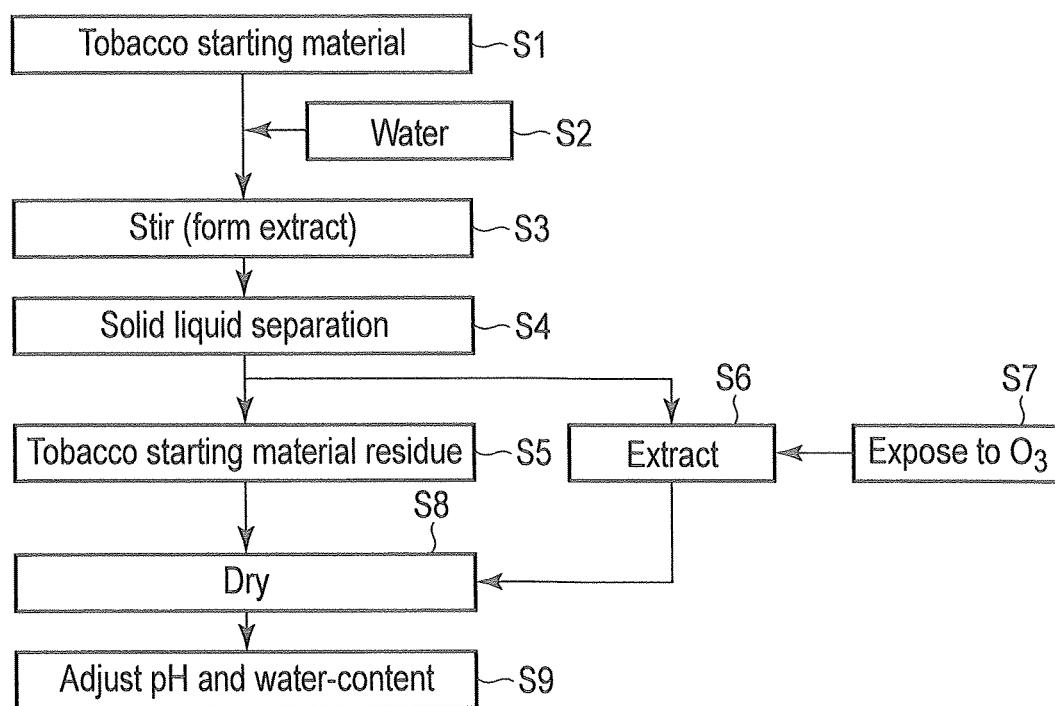


FIG. 3

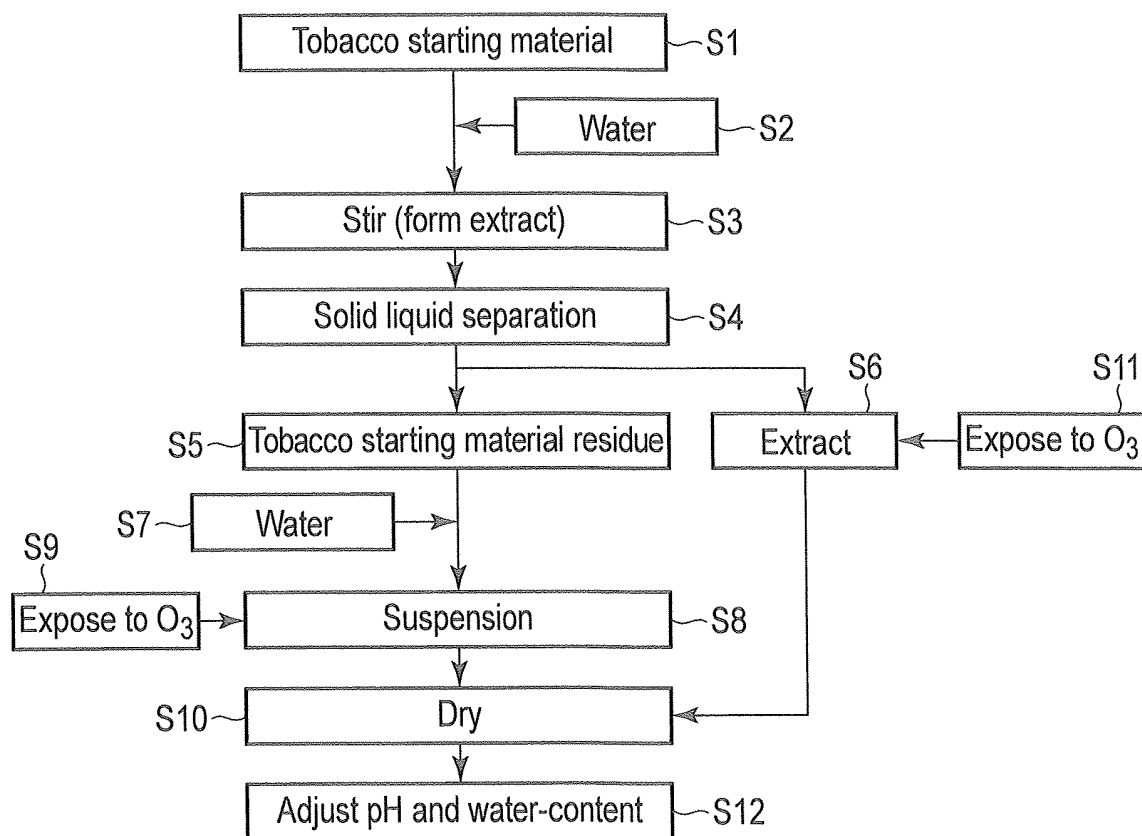


FIG. 4

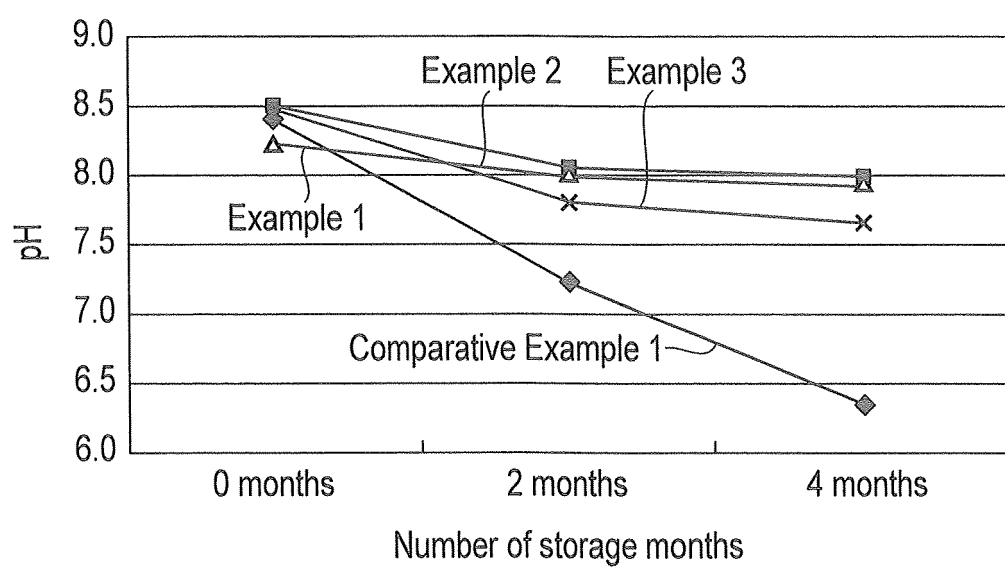


FIG. 5

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2013/059103

A. CLASSIFICATION OF SUBJECT MATTER

A24B15/24 (2006.01) i, A24B13/00 (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)

A24B15/24, A24B13/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2013

Kokai Jitsuyo Shinan Koho 1971-2013 Toroku Jitsuyo Shinan Koho 1994-2013

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X A	JP 2010-534475 A (R.J. Reynolds Tobacco Co.), 11 November 2010 (11.11.2010), paragraph [0133] & WO 2009/015142 A2 & CN 101873809 A	11-12 1-10
A	WO 2010/141278 A1 (R.J. REYNOLDS TABACCO CO.), 09 December 2010 (09.12.2010), & JP 2012-528593 A & CN 102802450 A	1-12
A	US 2006/0196516 A1 (Mingwu Cui), 07 September 2006 (07.09.2006), (Family: none)	1-12

☐ 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
17 June, 2013 (17.06.13)Date of mailing of the international search report
25 June, 2013 (25.06.13)Name and mailing address of the ISA/
Japanese Patent Office

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Form PCT/ISA/210 (second sheet) (July 2009)

REFERENCES CITED IN THE DESCRIPTION

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- JP 2009082331 PCT [0006]