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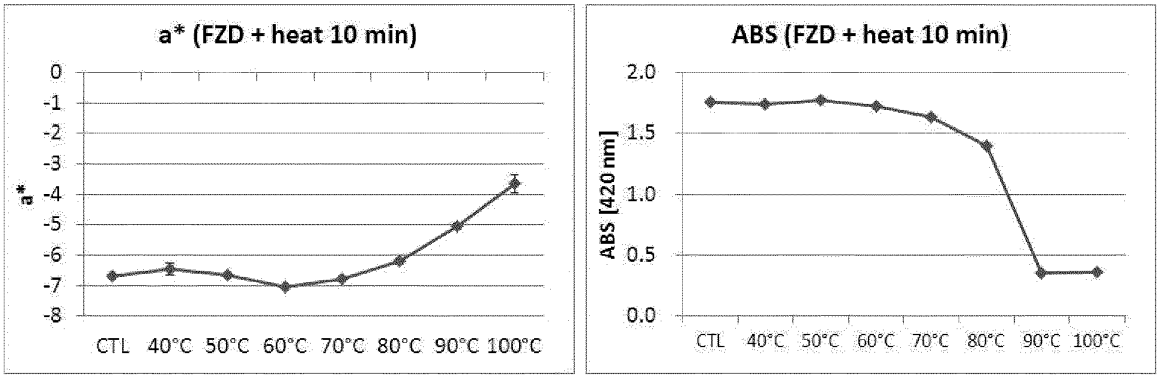
# (54) ORAL TOBACCO PRODUCT

(57) The problem addressed by the present invention is to provide an oral tobacco product such that the color of the tobacco raw material constituting the oral tobacco product maintains a green color without the color of the liquid extracted in a user's oral cavity becoming dark when the oral tobacco product is used. The oral tobacco product comprises a tobacco raw material which has an a* value, expressed by the L*a*b* method, of 1.0 or less and an extract of which, obtained using water as an extracting solvent, has a 420 nm wavelength light absorbance of 1.0 or less.

[Fig.2]




(a) (b)

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## Description

### TECHNICAL FIELD

**[0001]** The present invention relates to an oral tobacco product.

### BACKGROUND ART

**[0002]** In oral tobacco products such as snus, the tobacco raw material is contained in a packaging medium formed of a material such as nonwoven fabric. The user uses the product by placing it in the mouth.

**[0003]** Because oral tobacco products are used in this way, undesirable ingredients contained in the tobacco raw material sometimes seep out into the oral cavity. This problem is addressed in, for example, Patent Documents 1 to 3, which describe art for lowering the amount of nitrosamine contained in tobacco raw material when manufacturing oral tobacco products.

**[0004]** Patent Document 1 describes a method which, in order to reduce the amount of nitrosamine present in tobacco raw material, carries out treatment that includes the step of irradiating tobacco leaves with microwaves, thereby blocking the subsequent formation of nitrosamine in the tobacco leaves. Patent Document 2 discloses treatment for lowering the amount of nitrosamine by pressing the harvested tobacco leaves and carrying out steam treatment. Patent Document 3 discloses art in which air-dried, yellowed tobacco leaves are dried by a natural drying step and a controlled drying step, lowering the amounts of nitrite nitrogen and nitrosamine in the tobacco raw material.

**[0005]** Unlike the foregoing art, Patent Document 4 describes art relating to a smokeless cigarette composition and mentions a coloring agent as being included in the composition.

**[0006]**

Patent Document 1: Patent No. 4108754
Patent Document 2: Patent No. 3996958
Patent Document 3: Patent No. 3922985
Patent Document 4: Patent application laid-open No. 2009-508523

### DISCLOSURE OF THE INVENTION

**[0007]** All of the methods described in Patent Documents 1 to 3, given that green tobacco leaf is unsuitable for human consumption, carry out drying treatment (microwave or controlled drying) after the harvested tobacco leaf has lost its green color (after most of the tobacco has turned yellow). In the methods described in Patent Documents 1 to 3, the timing of the treatment of harvested tobacco leaf is disclosed to be at least after the tobacco is no longer in a green state.

**[0008]** However, the inventor has found that when this tobacco is used as the raw material for oral tobacco products, depending on the treatment conditions, the tobacco remains green even after treatment and extracts thereof which enter the oral cavity are light-colored.

**[0009]** In cases where, as in the method described in, for example, Patent Document 1, a tobacco raw material obtained by carrying out drying treatment on tobacco leaf that has changed color to yellow is used in an oral tobacco product, the color of extracts thereof which enter the oral cavity presumably darkens.

**[0010]** When an extract having such a dark color arises, this may have an undesirable impression on the user.

**[0011]** As noted above, in Patent Document 4, a coloring agent is mentioned as a material that is included in a smokeless cigarette composition. According to such art, a smokeless cigarette composition may be imparted with color, but the coloring action arises from the coloring agent.

**[0012]** Accordingly, the object of this invention is to provide an oral tobacco product in which the green color of the tobacco starting material is maintained without using a coloring agent, and which, when extraction is carried out using water as the solvent, gives a light-colored extract.

**[0013]** As a result of conducting extensive investigations, the inventor has found that, because an oral tobacco product containing a tobacco raw material obtained by a specific treatment process exhibits a green color and an extract obtained therefrom using water as the extracting solvent is light-colored, the above problems can be solved.

**[0014]** The invention is recited below.

[1] An oral tobacco product that includes a tobacco raw material which has an a* value, expressed by the L*a*b* method, of 1.0 or less and an extract of which, obtained using water as an extracting solvent, has a 420 nm wavelength light absorbance of 1.0 or less.

[2] The oral tobacco product according to [1], wherein the tobacco raw material is obtained by a treatment method including: a step of heating the tobacco raw material on condition that an enzyme eliciting enzymatic browning contained therein is deactivated; and a step of lowering the moisture content of the tobacco raw material, wherein tobacco raw material treatment is carried out after harvesting tobacco leaves serving as an starting material for the tobacco raw material and before the a* value expressed by the L*a*b* method becomes larger than 1.0 for 60% or more of surface areas of the harvested tobacco leaves.

[3] The oral tobacco product according to [2], wherein heating in the treatment method is carried out under conditions of from 80 to 100°C.

[4] The oral tobacco product according to [2] or [3], wherein the step of lowering the moisture content of the tobacco leaves in the treatment method is carried out before the step of carrying out heating, at a re-

duced pressure of less than 0.1 MPa and a temperature of 37°C or less.

[5] The oral tobacco product according to [2] or [3], wherein the step of lowering the moisture content of the tobacco leaves in the treatment method is carried out at the same time as or after the step of carrying out heating.

[6] The oral tobacco product according to any one of [2] to [5], wherein the step of carrying out heating is carried out by steam heating, microwave heating or gas-flow heating.

[7] The oral tobacco product according to any one of [1] to [6], wherein the proportion by weight of the tobacco raw material which has an a* value, expressed by the L*a*b* method, of 1.0 or less and an extract of which, obtained using water as the extracting solvent, has a 420 nm wavelength light absorbance of 1.0 or less accounts for 40 wt % or more of the overall tobacco raw material.

**[0015]** The invention provides an oral tobacco product for which the extract that enters the oral cavity of the user at the time of use is not dark-colored. In the oral tobacco product of the invention, the tobacco raw material making up the oral tobacco product retains a green color.

## BRIEF DESCRIPTION OF THE DRAWINGS

**[0016]**

FIG. 1A is graph showing the degree of greenness when conventional drying was carried out and when microwave drying was carried out using three types of tobacco leaf. FIG. 1B is a graph showing colorimetric values (ABS 420 nm) for extracts of tobacco raw materials obtained using the same starting materials and procedure as in FIG. 1A.

FIG. 2A is a graph showing colorimetric values (a* values) when tobacco leaf was heated at the various temperatures shown in the graph. FIG. 2B is a graph showing colorimetric values (ABS 420 nm) for extracts of tobacco raw materials obtained using the same starting materials and procedure as in FIG. 2A.

FIG. 3A is a graph showing colorimetric values (a* values) of various tobacco raw materials when tobacco leaf was heated under three temperature conditions and the heating time was varied. FIG. 3B is a graph showing colorimetric values (ABS 420 nm) for extracts of the various tobacco raw materials obtained by heating the same starting materials under the same heating conditions as in FIG. 3A.

FIG. 4A is a graph showing the colorimetric values (a* values) obtained after heat-treating harvested tobacco leaf under three differing conditions. FIG. 4B is a graph showing colorimetric values (ABS 420 nm) for extracts of the various tobacco raw materials obtained by heating the same starting materials under the same heating conditions as in FIG. 4A.

FIG. 5 presents graphs showing the relationship of the mixing ratio between the tobacco raw material of the invention and another tobacco raw material when these are mixed together versus the a* value (FIG. 5A) and versus the absorbance (ABS 420 nm) (FIG. 5B).

FIG. 6 shows an example of the points (shown as stars in the diagram) selected for tobacco leaf colorimetry (a* value) prior to treatment.

## MODE FOR CARRYING OUT THE INVENTION

**[0017]** The invention is described in detail below by way of embodiments, examples and the like. However, the invention is not limited to the following embodiments and examples, and may be practiced using any modifications thereto insofar as they do not depart from the spirit and scope of the invention.

<Tobacco Raw Material>

**[0018]** The type of tobacco serving as the tobacco raw material used in the oral tobacco product of the invention, although not particularly limited, is exemplified by the following genus *Nicotiana* varieties: the flue-cured and burley varieties of *N. tabacum,* and the brasilia variety of *N. rustica.*

**[0019]** The tobacco leaves used as the tobacco raw material, unlike in the inventions cited in the prior-art literature, are furnished to the treatment steps shown below before their color turns yellow; that is, when the leaves are for the most part green.

**[0020]** Here, the phrase "to turn yellow" means that a great majority (e.g., 60% or more, or even 90% or more) of the surface area of the harvested leaf has changed color to a degree where the a* value expressed by the L*a*b* method becomes a value larger than 1.0. The (green) pigment present in tobacco leaf decreases after the leaf has been harvested, causing the tobacco leaf to turn yellow. The a* value after harvesting of the tobacco leaf is generally from about -9 to about -1.5.

**[0021]** The tobacco raw material used in the oral tobacco product of the invention has an a* value (or greenness), expressed by the L*a*b* method, of 1.0 or less and an extract of this tobacco raw material, obtained using water as the extracting solvent, has a 420 nm wavelength light absorbance of 1.0 or less.

**[0022]** The tobacco raw material used in the oral tobacco product of the invention is not particularly limited so long as it satisfies the above conditions. Following harvest, the portion of the tobacco leaf from which the midrib has been removed may be furnished to the treatment operations. Alternatively, following harvest, the tobacco leaf may have moisture removed by pressing or the like, then be furnished to the treatment operations. Or, following harvest, the tobacco leaf may be refrigerated or frozen and stored, and subsequently furnished to the treatment operations.

**[0023]** The smaller the a* value in the L*a*b* method, the stronger the green color. The green color can be made stronger by harvesting the tobacco leaf at an earlier stage than when the tobacco leaf is to be used in ordinary cigarettes. Having the lower limit of the range in the a* value be -20 or above is suitable from the standpoint of ensuring the green color of the tobacco raw material. In cases where one wishes to make the green color of the tobacco raw material stronger, it is suitable to have the greenness (a* value) be -2 or below.

**[0024]** Measurement of the a* value of the tobacco raw material can be carried out by using a grinding mill to grind to a size of from 1 to 2 mm (mesh) tobacco raw material that has been dried to a moisture content of 3 to 5 wt %, and the color of the ground sample can be measured using a spectrophotometer. The color definitions are those expressed in the L*a*b* color space used by the Commission International de l'Eclairage (CIE) and JIS.

**[0025]** In the color measurement operation, a numerical result can be obtained by pouring the sample powder into a glass vessel to a layer thickness of 1 cm, directing a standard light (Standard Illuminant D65 for colorimetry, a standard illuminant defined by the CIE and the ISO) at the sample from the bottom of the vessel, and measuring the reflected light (reflected color measurement/specular component excluded (SCE) method).

**[0026]** The a* value can be adjusted by varying the heat treatment temperature and time in the subsequently described tobacco raw material treatment operations.

**[0027]** Specifically, a rise in the a* value can be suppressed by lowering the temperature or shortening the time of heat treatment.

**[0028]** Measurement of the a* value of the tobacco leaf prior to treatment is carried out as follows.

**[0029]** The color at 20 places on the surface of a tobacco leaf is measured using a spectrophotometer (KONICA MINOLTA/CM3500d, from Konica Minolta Holdings, Inc.). Color definitions, as with the above-described color measurement of the tobacco raw material, is expressed in the L*a*b* color system. The 20 places on the surface of the tobacco leaf are uniformly selected at, for example, the center and peripheral portions of the tobacco leaf, as indicated by the stars (★) in FIG. 6.

**[0030]** The color measurement operation is carried out by directing a standard light (Standard Illuminant D65 for colorimetry, a standard illuminant defined by the CIE and the ISO) at the sample and measuring the reflected light (reflected color measurement/specular component excluded (SCE) method), and the results are rendered into numerical values. A smaller a* value obtained upon measuring the tobacco leaf under these conditions may be regarded as indicating a greener color.

**[0031]** Also, an extract of the inventive tobacco raw material which is obtained using water as the extracting solvent has a 420 nm wavelength light absorbance (also indicated herein as "ABS 420 nm") of 1.0 or less.

**[0032]** In cases where the above treatment has been carried out on the tobacco raw material, this absorbance is generally 0.2 or more.

**[0033]** The extract is obtained by carrying out a procedure such as the following.

**[0034]** One part by weight of tobacco raw material obtained by lowering the moisture content to 5 wt % or less and grinding is weighed out and added to 25 parts by weight of 22°C water, and shaking extraction is carried out for about 10 minutes. The extract is then left to stand at the same temperature for about 20 minutes, after which it is filtered with a 0.20 μm membrane filter. The filtered extract is diluted two-fold with water and the 420 nm wavelength light absorbance is measured using an absorption spectrophotometer.

**[0035]** In this invention, the numerical value of the absorbance measured under these conditions is also called the "degree of browning"; a larger numerical value indicates a higher degree of brownness. For the purposes of this invention, "brown" and brown-colored" are synonymous.

**[0036]** This degree of browning can be adjusted by varying the heat treatment temperature and time in the subsequently described tobacco raw material treatment operation.

**[0037]** Specifically, it is possible to suppress an increase in the degree of browning by raising the heat treatment temperature or extending the heat treatment time.

<Oral Tobacco Product>

**[0038]** The oral tobacco product of the invention is exemplified by snus, gum, chewing tobacco, snuff, compressed tobacco (tablets, sticks, etc.), and edible films.

**[0039]** The oral tobacco product of the invention can be obtained by, for example, the following treatment method.

**[0040]** The tobacco raw material used in the oral tobacco product of the invention is obtained by a treatment method that includes the step of heating the tobacco raw material under conditions that deactivate an enzyme eliciting enzymatic browning contained therein and the step of lowering the moisture content of the tobacco raw material.

**[0041]** As mentioned above, by passing through a treatment method that includes the steps of heating under specific conditions and lowering the moisture content of the tobacco raw material, it is possible not only to keep the a* value of the resulting tobacco raw material low (i.e., retain the green color), but also to make the brownness of the extract smaller.

**[0042]** The prior art described above includes the step of, after the harvested tobacco leaf has turned yellow, drying the yellowed leaf. In such a case, the color of an extract of the tobacco raw material becomes darker. Specifically, in the case of tobacco raw material obtained via the step of drying (such as by microwave irradiation) the tobacco leaf after it has been harvested and has turned yellow, the extract has a colorimetric value (ABS 420 nm)

of typically about 1.4 or above.

**[0043]** By contrast, in this invention, as noted above, the treatment step is carried out before the color of the tobacco leaf turns yellow.

**[0044]** In this invention, "conditions that deactivate an enzyme eliciting enzymatic browning" refers to conditions such that the enzyme activity (polyphenyl oxidase (PPO) activity) value measured using the procedure described below becomes 0.02 U or less.

**[0045]** The enzyme activity value can be obtained by adding to an absorption spectrophotometer cell and mixing together a solution of enzyme protein extracted from the sample and a potassium phosphate buffer solution (pH 6.0), then adding to the mixture a 10 mM pyrocatechol solution as the substrate, and measuring at 40°C the increase in the 420 nm wavelength light absorbance with respect to a reference. A solution obtained by mixing in deionized water instead of the enzyme protein solution may be used as the reference.

**[0046]** After subtracting the increase in absorbance by the reference, the amount of enzyme that raises the absorbance of the sample ($\Delta$ABS) by 0.01 over a period of one minute is defined as 1 U.

**[0047]** In the above tobacco raw material treatment step, "conditions that deactivate an enzyme eliciting enzymatic browning" refers to conditions at which the enzyme activity (PPO activity) on the tobacco leaf becomes 0.02 U or less. An example of such conditions is heating at from 80 to 100°C. The heating time in this case is typically 40 minutes or less, and may be set according to the heating temperature within a range where the a* value does not undergo a large increase.

**[0048]** By passing through such heating, the enzyme eliciting enzymatic browning in the tobacco leaf is deactivated, as a result of which the tobacco raw material does not change color and remains green. In addition, an extract of the tobacco raw material can be made light-colored (having a small ABS 420 nm).

**[0049]** An embodiment in which such heating is carried out at 85°C or more for 10 minutes or less, and especially at 90°C or more for 5 minutes or less, is more preferred, both from the standpoint of the tobacco raw material not changing color and remaining green, and also from the standpoint of the color of the extract remaining light-colored.

**[0050]** The above-mentioned temperatures do not refer to the temperature setting of the heating apparatus, but rather to the temperature of the heated tobacco leaf itself.

**[0051]** The above heating step is exemplified by methods carried out by irradiation with microwaves or infrared light, methods carried out by applying steam, methods carried by immersion in hot water, methods carried out by applying hot air (gas-flow heating), and methods that involve direct contact with a medium such as heated metal. Of these, heating by gas-flow heating is preferred from the standpoint of convenience in production.

**[0052]** With regard to the above heating step, when raising the tobacco leaf to the desired temperature, it is preferable for the temperature to be elevated as quickly as possible and for the length of time that the temperature remains at the temperature levels (from 30°C to 85°C) during temperature rise to be as short as possible. This is to prevent the enzyme eliciting enzymatic browning within the tobacco leaf from working during temperature rise.

**[0053]** When the above heating is carried out by microwave irradiation, in one embodiment, microwave irradiation is carried out at, typically, a frequency of from about 900 to about 2,500 MHz.

**[0054]** Methods of heating by applying steam to the tobacco leaf are exemplified by methods in which superheated steam, which is vapor obtained by further heating saturated steam at the same pressure to give it a higher temperature than the saturation temperature, is applied to the tobacco leaf or the tobacco leaf is steamed at the saturated vapor pressure, which is a pressure (gauge pressure) of 0.1 MPa or more.

**[0055]** Methods of carrying out heating by directing hot air at the tobacco leaf are exemplified by the method of, for example, passing a stream of air having a temperature of 90°C or above and a relative humidity of 90% through the tobacco leaf for 10 minutes or less.

**[0056]** Methods of carrying out heat treatment by immersion in hot water are exemplified by boiling, in which the tobacco leaf is immersed in boiling water.

**[0057]** In addition to the above heating step, the operations carried out when producing tobacco raw material to be used in the oral tobacco product of the invention include, for example, the step of lowering the moisture content of the tobacco leaf.

**[0058]** Including not only the above heating step but also the step of lowering the moisture content helps both to maintain the greenness of the tobacco raw material and also to maintain the light color of the tobacco raw material extract.

**[0059]** Methods of lowering the moisture content of the tobacco leaf include pneumatic drying and freeze drying in which a low-temperature, normal-temperature or high-temperature stream of air is applied to the tobacco leaf which is either at rest or fluidized, drying by irradiation with microwaves or infrared light, and drying by direct contact with a medium such as heated metal.

**[0060]** The same conditions and apparatus as when carrying out the above-described heating may be used for drying by the application of a high-temperature stream of air (hot air), drying by means of microwaves or infrared light, and drying by direct contact with a medium such as heated metal.

**[0061]** The above step in which heating is carried out and the above step in which the moisture content of the tobacco leaf is lowered are both essential for preparing the tobacco raw material of the invention.

**[0062]** Exemplary combinations in which these steps may be carried out are shown below.

1. Heating before Drying: For example, gas-flow heating is carried out as the heating step, following which freeze-drying is carried out.
2. Heating during Drying: In this embodiment, heating of the tobacco raw material and lowering of the moisture content are carried out at the same time by carrying out, for example, microwave irradiation.
3. Heating after Drying: In this embodiment, the moisture content of the tobacco leaf is lowered, after which heating is carried out (e.g., heating by a method that maintains the real temperature of the tobacco at 80°C or above for 10 minutes or less, such as condensation heat transfer by heated steam, convective heat transfer by a stream of air, or conductive heat transfer by contact with a high-temperature object such as metal or plastic).

**[0063]** Regardless of which of the above Methods 1 to 3 is carried out, the greenness of the resulting tobacco raw material is maintained and the degree of browning by the extract becomes smaller.

**[0064]** Moreover, regardless of which of the above Methods 1 to 3 has been used, the moisture content of the tobacco raw material becomes 20 wt % or less, preferably 10 wt % or less, and more preferably 5 wt % or less.

**[0065]** When Method 1 above is carried out, in a preferred embodiment, the harvested tobacco leaf is subjected to heating at 85°C or above for 10 minutes or less, following which the moisture content of the tobacco raw material is lowered using a known freeze-drying method.

**[0066]** When Method 2 above is carried out, in a preferred embodiment, the harvested tobacco leaf is irradiated with microwaves so as to bring the tobacco leaf up to 85°C or above, thereby heating and lowering the moisture content for 10 minutes or less.

**[0067]** When Method 3 above is carried out, in a preferred embodiment, the operation of lowering the moisture content of harvested tobacco leaf entails, for example, carrying out freeze-drying so as to bring the moisture content of the tobacco raw material down to 20 wt % or less, preferably 10 wt % or less, and even more preferably 5 wt % or less; then carrying out heating for 10 minutes or less at an actual tobacco leaf temperature of 85°C or more via, for example, condensation heat transfer by heated steam, convective heat transfer by a stream of air, or conductive heat transfer by contact with a high-temperature object such as metal or plastic.

**[0068]** When Method 3 above is carried out, to suppress a decrease in the green pigment contained in the tobacco leave, it is preferable to carry out the step of lowering the moisture content at a pressure of less than 0.1 MPa and at 37°C or below. In the moisture content lowering step, the pressure is more preferably 0.05 MPa or less, and even more preferably 0.01 MPa or less. This step is more preferably carried out at a temperature of 0°C or less.

**[0069]** In each of the above embodiments, the tobacco leaf used in treatment is used after being harvested and before the a* value (greenness) becomes larger than 1.0.

**[0070]** The tobacco raw material used in the oral tobacco product of the invention is obtained by treatment that includes, as described above, the step of heating and the step of lowering the moisture content. Were this a question of merely raising the greenness (a* value) of the tobacco raw material after drying, carrying out freeze-drying alone would suffice. However, tobacco raw material obtained by carrying out freeze-drying alone incurs a pronounced decrease in quality after drying and, together with moisture absorption after drying, undergoes browning.

**[0071]** Moreover, extracts of tobacco raw materials that have only been freeze-dried undergo pronounced browning. In this case, the essential features of the invention, namely that the tobacco raw material is green in color and that an extract thereof has a low degree of browning, are not satisfied.

**[0072]** Deactivating an enzyme eliciting enzymatic browning by passing through the step of carrying out the above-described heating is effective as one condition for keeping the extract from turning brown.

<Oral Tobacco Product>

**[0073]** In cases where the oral tobacco product of the invention is made into, for example, snus, this product is obtained by using a known method to fill the above-described tobacco raw material into a packaging medium formed of a material such as nonwoven fabric. For example, the snus is obtained by carrying out filling while adjusting the amount of tobacco raw material, and by carrying out sealing using a means such as heat-sealing.

**[0074]** The packaging medium may be used without any particular limitation, although preferred use may be made of a cellulose-based nonwoven fabric.

**[0075]** In cases where the oral tobacco product of the invention is a gum, for example, this gum may be obtained by employing a known method to mix the above-described tobacco raw material used in this invention together with a known gum base. In the case of chewing tobacco, snuff and compressed tobacco as well, aside from using the tobacco raw material described in this invention, these may be obtained by known methods.

**[0076]** In the oral tobacco product of the invention, the proportion by weight of the total amount of tobacco raw material accounted for by the tobacco raw material which has an a* value expressed by the L*a*b* method of 1.0 or less and an extract of which, obtained using water as the extracting solvent, has a 420 nm wavelength light absorbance of 1.0 or less may be suitably adjusted, but is preferably as large as possible, with a proportion of 40 wt % or more being preferred, a proportion of 60 wt % or more being more preferred, a proportion of 80 wt % or more being even more preferred, a proportion of 98 wt % or more being still more preferred, and a proportion of 100 wt % being most preferred.

**[0077]** In cases where tobacco raw materials other

than the above-described specific tobacco raw material are included in the oral tobacco product of the invention, use is made of tobacco raw materials which do not detract from the advantageous effects of the invention.

**[0078]** In the oral tobacco product of the invention, as explained above, because the degree to which the tobacco raw material extract turns brown is small, the seepage of brown color to the packaging medium is likely to be suppressed. Also, the color of the tobacco raw material is kept green.

EXAMPLES

**[0079]** The invention is described more fully below by way of examples. However, the invention, insofar as it does not depart from the spirit and scope thereof, is not limited to the following examples.

**[0080]** Hereinafter, the invention is described more fully below by way of examples. However, the invention, insofar as it does not depart from the spirit and scope thereof, is not limited to the following examples.

<Experimental Example 1-1> Influence of Differences in Leaf Tobacco Drying Method (Conventional Drying and Microwave Drying)

**[0081]** An experiment was carried out in which three varieties of tobacco leaf were subjected to conventional drying or microwave drying, and the appearance (perceived color) of the dried leaves was measured. The varieties of raw material were respectively Burley (BLY), flue-cured tobacco (FCV), and *N. rustica* (Rustica).

**[0082]** The perceived color of the appearance was measured by the following procedure.

**[0083]** A tobacco raw material that was dried to a moisture content of 3 to 5% was ground to a size of 1 to 2 mm mesh using a grinding mill (MiniBlender, from Melitta Japan, Ltd. (Tokyo, Japan)), and the color of the ground sample was measured using a spectrophotometer (KONICA MINOLTA/CM3500d, from Konika Minolta Holdings, Inc.). The color definitions were expressed in the L*a*b* color system used by the Commission International de l'Eclairage (CIE) and JIS.

**[0084]** In the color measurement operation, numerical results were obtained by pouring the sample powder into a glass vessel to a layer thickness of 1 cm, directing a standard light (Standard Illuminant D65 for colorimetry, a standard illuminant defined by the CIE and the ISO) at the sample from the bottom of the vessel, and measuring the reflected light (reflected color measurement/specular component excluded (SCE) method).

**[0085]** The results are shown in FIG. 1A.

**[0086]** The vertical axis (a*) in FIG. 1A shows the spectrophotometer data expressed by the L*a*b* method; a smaller a* value indicates a greener color. On comparing the color measurement values, for all three types of raw materials, the microwave-dried leaf had a higher degree of greenness than the conventionally dried tobacco leaf.

**[0087]** Moreover, in this invention, the following drying methods were used as conventional drying, depending on the variety of tobacco leaf. The following conventional drying methods were all carried out under normal pressure.

**[0088]** In the case of flue-cured tobacco, the harvested tobacco leaf is dried by a step called "flue-curing." Generally, after being harvested, the leaf is cured for 3 days in a humidity-controlled drying room at 30 to 50°C, following which it is dried for 2 days at about 70°C, thereby lowering the moisture in the tobacco raw material to 5 wt % or less.

**[0089]** In the case of Burley tobacco, the harvested tobacco leaf is dried by a step called "air-curing." Generally, after being harvested, the leaf is dried to a moisture content of 15 wt % or less over a period of 30 to 35 days in a humidity-controlled environment at from the outside temperature to 35°C.

**[0090]** In the case of *N. rustica,* the harvested tobacco leaf is dried by a step called "sun-air-curing." Generally, after being harvested, the leaf is sun-dried at the outside temperature for a period of about several days to one week.

<Experimental Example 1-2>

**[0091]** The colorings of extracts obtained from the same three types of raw materials as in Experimental Example 1-1 were measured. The results are shown in FIG. 1B. The colors of the extracts, as explained above, are expressed by the 420 nm wavelength light absorbance. A higher absorbance (larger Y-axis value) indicates a browner color.

**[0092]** The degree of browning for the tobacco raw material extracts was measured by the following method. Raw material (tobacco leaf) dried to a moisture content of 5% or less was weighed out in an amount of 0.4 g, then 10 mL of water was added and shaking extraction was carried out for 10 minutes at 22°C. After shaking, the extract was left to stand at 22°C for 20 minutes, then was filtered with a 0.20 μm pore diameter membrane filter (Whatman PVDF membrane, from GE Healthcare UK, Ltd. (Buckinghamshire, UK)). The resulting solution was diluted two-fold with water, and the 420 nm wavelength light absorbance was measured using a spectrophotometer.

**[0093]** As shown in FIG. 1B, for each of the three types of raw material, extracts of tobacco raw materials obtained by microwave drying had a lower degree of browning than did extracts of tobacco raw material obtained by conventional drying.

**[0094]** That is, compared with cases in which a conventionally dried tobacco raw material is used as the starting material in an oral tobacco product, when a microwave-dried tobacco raw material is used as the starting material in an oral tobacco product, one can expect the color of saliva in the mouth to be lighter and the nonwoven fabric making up the packaging medium in the

oral tobacco product to not become brown-colored following use.

<Experimental Example 2> Various Effects at Respective Heating Temperature

**[0095]** Heat treatment was carried out on the tobacco leaf, and the appearance (perceived color) of the tobacco leaf at that time as well as the appearance of the extract (perceived color) were measured.

**[0096]** The tobacco leaf used here was a freeze-dried Burley.

**[0097]** Heat Treatment: Heating was carried out at from 40 to 100°C for 10 minutes. The tobacco leaf used as the starting material was vacuum-packed, then was heat-treated in a hot-water bath. The results are shown in FIGS. 2A and 2B. It is apparent from these results (the colorimetric values when the tobacco leaf (the leaf obtained by freeze-drying Burley tobacco) was heated at the respective temperatures for 10 minutes) that the greenness decreases with heating at 80°C or above (FIG. 2A). On the other hand, with heating to 85°C or above, the color of an extract of the heat-treated tobacco raw material becomes lighter (the degree of browning becomes lower) (FIG. 2B).

<Experimental Example 3> Various Effects at Respective Heating Times

**[0098]** Heat treatment was carried out on tobacco leaf, and the appearance (perceived color) at that time as well as the appearance (perceived color) of extracts thereof were measured by the same procedure as in Experimental Example 1.

**[0099]** The types of tobacco leaf used were Burley and freeze-dried tobacco.

**[0100]** Heat treatment conditions: Three levels of 80, 90 and 100°C, a heating time of from 1 to 40 minutes, and the same heating method as in Experimental Example 2.

**[0101]** FIG. 3A shows the color measurement values for the treated raw material when tobacco leaf (freeze-dried Burley leaf) was heat-treated under three conditions - 80°C, 90°C and 100°C - for varying heat treatment times. At each of these temperatures, the degree of greenness decreased with the passage of heating time. At a higher heating temperature, the color faded in a shorter time.

**[0102]** FIG. 3B shows the degree of browning of extracts of the treated raw material when tobacco leaf (freeze-dried Burley leaf) was heat-treated under three conditions - 80°C, 90°C and 100°C - for varying heat treatment times. With the passage of the heating time, the degree of browning of the treated raw material was found to be increasingly suppressed.

**[0103]** From these results, although the degree of brownness of the extract was substantially the same (FIG. 3B) with 30 minutes of heating at 80°C (A), 5 minutes at 90°C (B), and 2 minutes of heating at 100°C (C), the degree of greenness in these cases differed completely (FIG. 3A).

<Experimental Example 4> Timing of the Heating Step

**[0104]** The appearance of the tobacco raw material (FIG. 4A) and the appearance of the tobacco raw material extract (FIG. 4B) were examined when heat treatment was carried out at various times-namely, before, during or after drying of the tobacco leaf.

**[0105]** The tobacco leaf used was Burley tobacco, and the treatment method used in the examples was carried out as follows.

- Heating before Drying: The harvested leaves were gas-flow heated (85°C, 10 minutes), then freeze-dried.
- Heating during Drying: The harvested leaves were heated and dried by microwave heating
- Heating after Drying: The harvested leaves were freeze-dried, then heated at 100°C for 3 minutes
- Control: Raw material obtained by conventional heating of Burley (air-cured leaf)

**[0106]** From the results shown in FIGS. 4A and 4B, it was found that, regardless of whether heat treatment is carried out before, during or after drying, so long as certain fixed conditions are satisfied, the greenness of the tobacco raw material can be ensured and discoloration (darkening in the color) of extracts of the tobacco raw material can be prevented. The fixed conditions are that heat treatment and drying be completed before the leaves change color and, as in Experimental Example 3, that heat treatment be carried out at a high temperature and for a short period of time.

<Experimental Example 5>

**[0107]** The relationship between the a* value and the absorbance (420 nm) obtained when the tobacco raw material of the invention (a* value, -11.0; 420 nm wavelength light absorbance of extract, 0.3) and another tobacco raw material (a tobacco raw material typically used in oral tobacco products; a* value, 8.31; 420 nm wavelength light absorbance of extract, 1.4) are mixed together was measured.

**[0108]** The results are shown in FIGS. 5A and B. The horizontal axis in these graphs represent the proportion (wt %) of tobacco raw material of the invention.

**[0109]** In cases where the tobacco raw material of the invention and another, ordinary, tobacco raw material are mixed together, at a proportion of generally 40 wt % or more, a desirable a* value and absorbance (420 nm) can be obtained.

<Reference Example 1> Yellowing of Harvested Tobacco Leaf

**[0110]** Flue-cured (FCV) tobacco leaf that had been stored until most of the leaf surface turned yellow was measured with a colorimeter. As a result, the greenness (a* value) of the portion that turned yellow, which accounts for the majority of the leaf surface, was 2.0±0.7. When this tobacco leaf was microwave dried and rendered into yellow tobacco raw material, the a* value was 5.3±0.1, and the absorbance (ABS 420 nm) was 1.57.

**[0111]** When change has occurred so that the great majority of the tobacco leaf surface (60% or more of the surface area) has a greenness (a* value) larger than 1.0, the tobacco leaf is referred to as "leaf that has turned yellow"; in other words, the advantageous effects of the invention are not obtained unless at least the great majority of the tobacco leaf (60% or more of the surface area) has an a* value that is 1.0 or less.

**[0112]** The degrees of greenness (a* value) of the tobacco leaf (frozen tobacco leaf) prior to drying of the Burley, flue-cured and *N. rustica* tobacco used in the experimental examples of the invention were respectively as follows: Burley, - 2.1±0.3; flue-cured, -2.5±0.6; *N. rustica,* -4.6±0.5.

<Reference Example 2>

(Method of Measuring Activity of Enzyme eliciting enzymatic browning in Tobacco Raw Material)

**[0113]** An experiment was carried out in which, for tobacco raw material, to ascertain the desirable effects of including a heating step, the enzyme eliciting enzymatic browning activities in tobacco leaf that was not heat-treated (a Burley variety (Michinoku) which was subjected only to freeze-drying, a Burley variety (Burley 21) which was subjected only to freeze-drying) and in tobacco raw material that had passed through the steps of heating and moisture reduction according to this invention were measured.

**[0114]** Measurement of the enzyme eliciting enzymatic browning (PPO) activities in the respective tobacco raw materials was carried out as follows. Tobacco leaf was dried to a moisture content of 5 wt % or less, 10 mL of a 20 mM potassium phosphate buffer (pH 6.0) was added to 200 mg of the ground sample, and the mixture was homogenized for 2 minutes in an ice-cooled environment. This was followed by 10 minutes of ultrasonication in an ice-cooled environment, thereby extracting enzyme protein. This solution was filtered using a membrane filter made of a cellulose acetate filter material having a pore diameter of 0.20 μm (ADVANTEC Cellulose Acetate filter). PPO activity measurement was carried out using this enzyme protein solution, a 0.1 M potassium phosphate buffer solution (pH 6.0), and a pyrocatechol solution serving as the substrate. The activity measurement procedure was as follows. A 0.1 M potassium phosphate buffer solution (900 μL) and 100 μL of an enzyme protein extract prepared by the method described above were mixed together within an absorption spectrophotometer cell. As the control, 900 μL of a 0.1 M potassium phosphate buffer solution and 100 μL of deionized water were mixed together, and this was used as a reference. In an environment set to 40°C, 1.00 mL of a 10 mM pyrocatechol solution was mixed with the sample and the reference, and the increase in 420 nm wavelength light absorbance with respect to the reference was observed at 40°C. Measurement involved measuring the rate of increase in absorbance starting immediately after addition of the substrate and up until 15 seconds to 60 seconds after addition.

**[0115]** The enzyme activities of tobacco leaf that had not been heat-treated were 4.64 U (freeze-dried leaf of a Burley variety (Michinoku)) and 6.42 U (freeze-dried leaf of a Burley variety (Burley 21)), whereas the enzyme activity of tobacco raw material that passed through the heating and moisture-lowering steps were all 0.02 U or less. Based on these results, the heat treatment of tobacco leaf can be regarded as effective for deactivating PPO.

## INDUSTRIAL APPLICABILITY

**[0116]** Because the tobacco raw material included in the oral tobacco product of the invention retains the green color of the tobacco leaf after harvesting and extracts thereof exhibit a light color, discoloration due to seepage of the oral tobacco product color into the mouth of the user can be prevented.

## Claims

1. An oral tobacco product comprising a tobacco raw material which has an a* value, expressed by the L*a*b* method, of 1.0 or less and an extract of which, obtained using water as an extracting solvent, has a 420 nm wavelength light absorbance of 1.0 or less.

2. The oral tobacco product according to claim 1, wherein the tobacco raw material is obtained by a treatment method including: a step of heating the tobacco raw material on condition that an enzyme eliciting enzymatic browning contained therein is deactivated; and a step of lowering the moisture content of the tobacco raw material, wherein
the tobacco raw material treatment is carried out after harvesting tobacco leaves serving as a starting material for the tobacco raw material and before the a* value expressed by the L*a*b* method becomes larger than 1.0 for 60% or more of surface areas of the harvested tobacco leaves.

3. The oral tobacco product according to claim 2, wherein heating in the treatment method is carried

out under conditions of from 80 to 100°C.

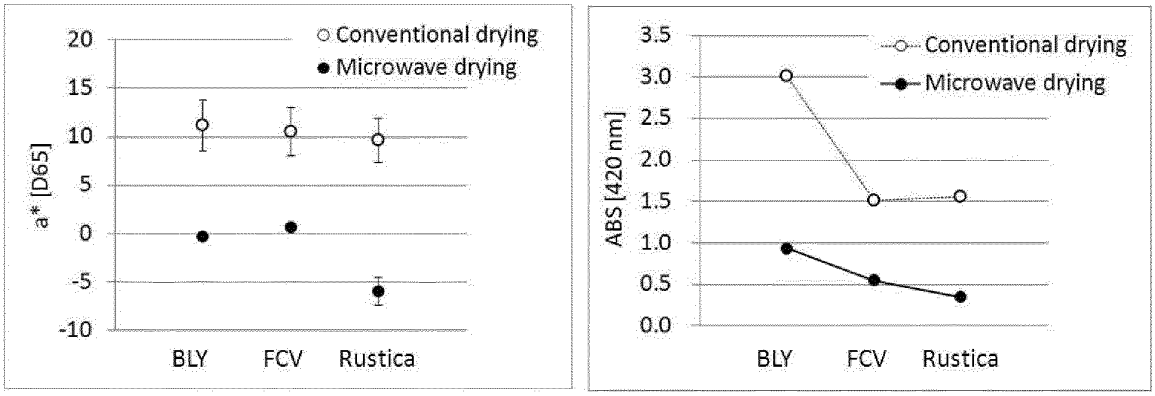
4. The oral tobacco product according to claim 2 or 3, wherein the step of lowering the moisture content of the tobacco leaves in the treatment method is carried out before the step of carrying out heating, at a reduced pressure of less than 0.1 MPa and a temperature of 37°C or less.

5. The oral tobacco product according to claim 2 or 3, wherein the step of lowering the moisture content of the tobacco leaves in the treatment method is carried out at the same time as or after the step of carrying out heating.

6. The oral tobacco product according to any one of claims 2 to 5, wherein the step of carrying out heating is carried out by steam heating, microwave heating or gas-flow heating.

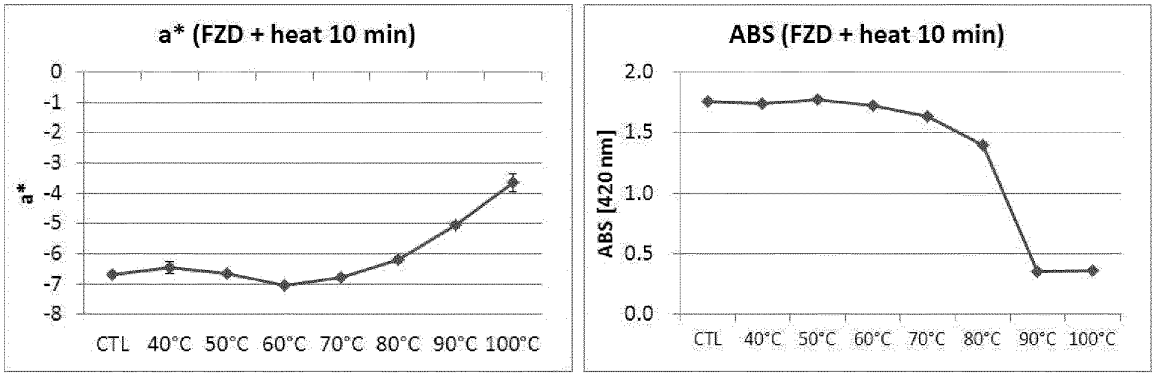
7. The oral tobacco product according to any one of claims 1 to 6, wherein the proportion by weight of the tobacco raw material which has an a* value, expressed by the L*a*b* method, of 1.0 or less and an extract of which, obtained using water as an extracting solvent, has a 420 nm wavelength light absorbance of 1.0 or less accounts for 40 wt % or more of the overall tobacco raw material.

[Fig.1]



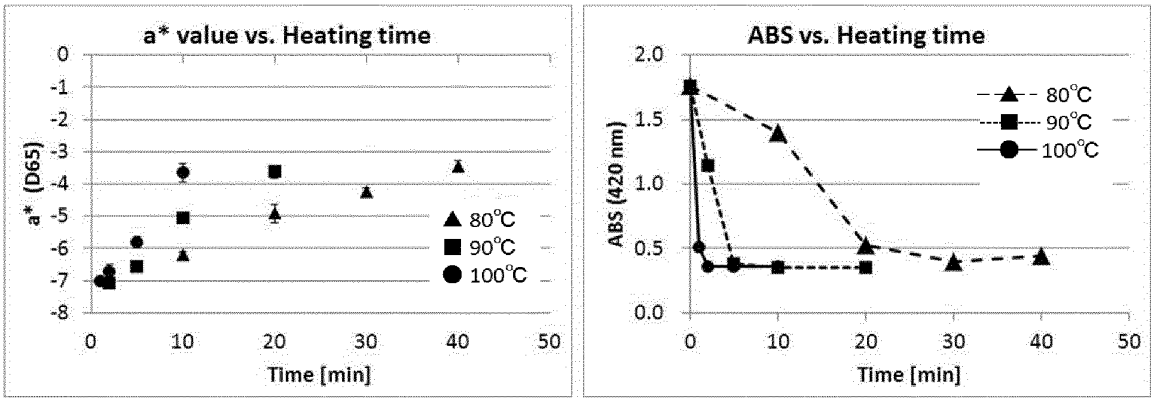

(a) (b)

[Fig.2]



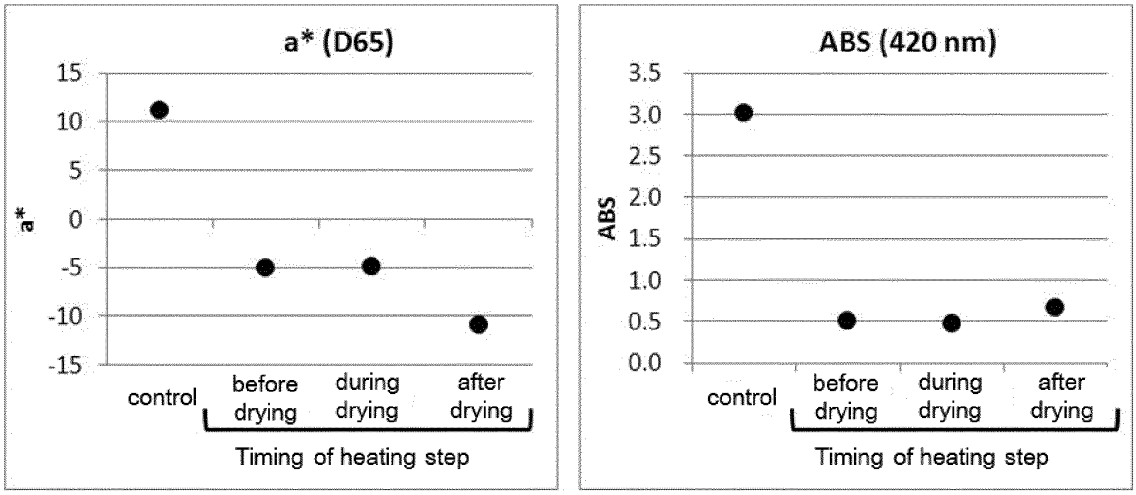

(a) (b)

[Fig.3]



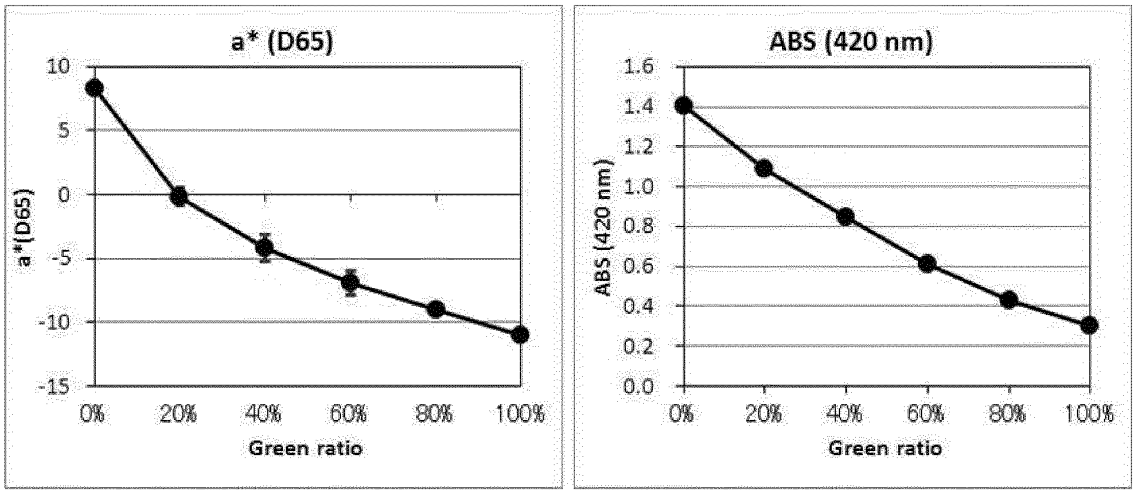

(a) (b)

[Fig.4]



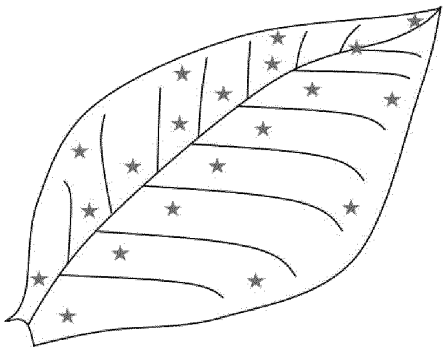

(a) (b)

[Fig.5]




(a) (b)

[Fig.6]



**INTERNATIONAL SEARCH REPORT**

International application No.
PCT/JP2014/054497

A. CLASSIFICATION OF SUBJECT MATTER
*A24B13/00*(2006.01)i, *A24B3/04*(2006.01)i, *A24B3/12*(2006.01)i, *A24B15/24*(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)
A24B13/00, A24B3/04, A24B3/12, A24B15/24

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 1996-2014
Kokai Jitsuyo Shinan Koho 1971-2014 Toroku Jitsuyo Shinan Koho 1994-2014

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. |
|---|---|---|
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| A | JP 2012-1 A (Japan Tobacco Inc.), 05 January 2012 (05.01.2012), entire text; all drawings & WO 2010/041659 A1 | 1-7 |
| A | JP 2010-521957 A (Philip Morris Products S.A.), 01 July 2010 (01.07.2010), entire text; all drawings & US 2008/0202536 A1 & WO 2008/104891 A2 | 1-7 |

☒ 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 | Date of mailing of the international search report |
|---|---|
| 10 April, 2014 (10.04.14) | 22 April, 2014 (22.04.14) |
| Name and mailing address of the ISA/ Japanese Patent Office | Authorized officer |
| Facsimile No. | Telephone No. |

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**INTERNATIONAL SEARCH REPORT**

International application No.
PCT/JP2014/054497

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
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## REFERENCES CITED IN THE DESCRIPTION

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