(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 2 818 059 A1

(12) **EUROPEAN PATENT APPLICATION**
published in accordance with Art. 153(4) EPC

(43) Date of publication:
31.12.2014 Bulletin 2015/01

(21) Application number: **13752001.1**

(22) Date of filing: **20.02.2013**

(51) Int Cl.:
A24B 13/00 *(2006.01)* ***A24B 15/32*** *(2006.01)*

(86) International application number:
PCT/JP2013/054195

(87) International publication number:
WO 2013/125586 (29.08.2013 Gazette 2013/35)

(84) Designated Contracting States:
AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR
Designated Extension States:
BA ME

(30) Priority: **24.02.2012 JP 2012039028**

(71) Applicant: **JAPAN TOBACCO INC.**
Minato-ku
Tokyo 105-8422 (JP)

(72) Inventors:
- **YOKOI, Michinori**
  Tokyo 130-8603 (JP)
- **FURUKOSHI, Masashi**
  Tokyo 130-8603 (JP)

(74) Representative: **Isarpatent**
Patent- und Rechtsanwälte
Friedrichstrasse 31
80801 München (DE)

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

(57) A method for producing an oral tobacco material, characterized by comprising the steps of: adding an antioxidant to a tobacco raw material consisting of a dry oral tobacco material having a pH of 8 or less; and heating the antioxidant-added tobacco raw material at a temperature of 80°C or higher.

EP 2 818 059 A1

Description

Technical Field

[0001] The present invention relates to a method for producing an oral tobacco material, and an oral tobacco material. More specifically, the present invention relates to a method for producing an oral tobacco material having a reduced content of tobacco-specific nitrosamine, and an oral tobacco material obtained by that production method.

Background Art

[0002] Recently, oral tobacco products including snus have been drawing attention. The oral tobacco product contains an oral tobacco material such as powdered tobacco in a water-permeable pouch. People insert such an oral tobacco product between a lip and a gum in a mouth, and enjoy the taste and flavor of the powdered tobacco (see, for example, Patent Document 1).

[0003] The oral tobacco material used in such an oral tobacco product contains various components such as nicotine, nitrosamines (including tobacco-specific nitrosamine), hydrocarbons, and proteins, similar to a general tobacco material for a cigarette. In these components, there are some components which desirably are not removed or are increased in terms of smoking taste or other reasons; whereas, there are also some components whose amounts are desirably decreased. It is desired to decrease the amount of the tobacco-specific nitrosamine.

Prior Art Document

Patent Document

[0004] Patent Document 1: U.S. Patent Application Publication No. 2007/062549

Summary of Invention

Problem to be solved by the Invention

[0005] The object of the present invention is to provide a method for producing an oral tobacco material having a reduced content of tobacco-specific nitrosamine (TSNA).

Means for solving the Problem

[0006] In order to solve the problem, according to the first aspect of the present invention, there is provided a method for producing an oral tobacco material, characterized by comprising the steps of: adding an antioxidant to a tobacco raw material consisting of a dry oral tobacco material having a pH of 8 or less; and heating the antioxidant-added tobacco raw material at a temperature of 80°C or higher.

[0007] Further, according to the second aspect of the present invention, there is provided an oral tobacco material which is obtained by the method for producing an oral tobacco material according to the present invention.

Effects of the Invention

[0008] The oral tobacco material produced according to the present invention shows a significantly reduced TSNA content.

Description of Embodiments

[0009] Various embodiments of the present invention will be explained in detail below.

[0010] In the method for producing an oral tobacco material according to the present invention, a dry oral tobacco material having a pH of 8 or less is used as a raw material. A dry oral tobacco material having a pH of 4 or more is usually used as the raw material. In the present specification, the pH of a dry tobacco material refers to a value obtained by adding 20 to 100 g of distilled water (corresponding to 10 times the amount of the tobacco material) to 2 to 10 g of the tobacco material; shaking the mixture of the water and the tobacco material at 200 rpm at room temperature for 10 minutes; allowing the resulting mixture to stand for 5 minutes; and then measuring the pH of an extract obtained therefrom with a pH meter (IQ 240 manufactured by IQ Scientific Instruments Inc.).

[0011] The tobacco raw material includes a dry oral tobacco material which originally has a pH of 8 or less, and a

material obtained by adding an acidic substance to a dry oral tobacco material having a pH of more than 8 to adjust the pH to 8 or less.

[0012] The former, the dry oral tobacco material which originally has a pH of 8 or less may include flue-cured tobacco, burley tobacco, dark air-cured tobacco, dark fire-cured tobacco, air-cured tobacco by early-dehydrating, sun-cured tobacco, oriental tobacco, and the like.

[0013] The latter, the dry oral tobacco material having a pH of more than 8 may include a part of burley tobacco, dark air-cured tobacco, air-cured tobacco, and the like. As the acidic substance added to the above tobacco material, an organic acid such as citric acid, ascorbic acid, acetic acid, lactic acid, malic acid, tartaric acid, or succinic acid may be used. A dry oral tobacco material exhibiting acidity (i.e., a pH of less than 7) (for example, flue-cured tobacco, burley tobacco, dark air-cured tobacco, dark fire-cured tobacco, air-cured tobacco by early-dehydrating, sun-cured tobacco or oriental tobacco) may also be used, instead of the organic acid. When the dry oral tobacco material exhibiting acidity is used as the acidic substance, the amount thereof is preferably 100% by weight or less with respect to the raw material.

[0014] Next, an antioxidant is added to the tobacco raw material described above. The antioxidant significantly decreases the amount of TSNA contained in the tobacco raw material. TSNA typically includes N'-nitrosonornicotine (NNN), 4-(methylnitrosoamino)-1-(3-pyridyl)-1-butanone (NNK) and N'-nitrosoanatabine (NAT). The antioxidant may include epigallocatechin gallate, ascorbic acid, green tea extract, and the like. Ascorbic acid is also the acidic substance for adjusting the pH, as described above; and it may be used as the antioxidant in combination with another acidic substance, or may be used alone to serve both as the antioxidant and the acidic substance. The antioxidant is added preferably in an amount of 1 to 100 parts by weight based on 100 parts by weight of the tobacco raw material.

[0015] Next, the tobacco raw material to which the antioxidant has been added is heated at a temperature of 80°C or higher. The heating temperature is usually 130°C or lower. The heating time is usually from 10 minutes to 30 minutes.

[0016] In order to heat the tobacco raw material at a temperature of higher than 100°C, for example, the tobacco raw material is put in a closed heating container (such as a cylindrical one) having a jacket at the outer side thereof and equipped with an introduction pipe and an exhaust pipe of heated steam, and heated steam is directly blown into the tobacco raw material through the introduction pipe, while the heated steam is circulated in the jacket, whereby the tobacco material can be heated at the same temperature as that of the heated steam.

[0017] Nitrite salts contained in the tobacco raw material become unstable when the tobacco raw material has a pH of 8 or less, and the nitrite salts can be decomposed and released as NO_x gas by heating the tobacco raw material at 80°C or higher. Therefore, according to the method of the present invention, the amount of TSNA contained in the tobacco raw material can be decreased and the amount of the nitrate salt can also be decreased.

Examples

[0018] The present invention will be explained by means of Examples, but the invention is not limited to those Examples.

Example 1

[0019] 5 kg of ground dry oral tobacco material (pH of 8.3) was put in a heating container, and 2% by weight of citric acid and 2% by weight of epigallocatechin gallate were added thereto, thereby adjusting the pH to 6.1. While the pH-adjusted tobacco material was stirred, heated steam having a temperature of 110°C was blown into the tobacco material and also the heated steam having a temperature of 110°C was circulated in a jacket provided at the outer side of the heating container, whereby the tobacco material was heated at 110°C for 30 minutes. After that, the heating container was cooled to room temperature, and the tobacco material was taken out from the container. A nitrous acid concentration and a TSNA concentration in the tobacco material were measured by the following methods. The results are shown in Table 1.

Example 2

[0020] 5 kg of ground dry oral tobacco material (pH of 8.3) was put in a heating container, and 2% by weight of citric acid and 2% by weight of a green tea extract were added thereto, thereby adjusting the pH to 6.3. While the pH-adjusted tobacco material was stirred, heated steam having a temperature of 110°C was blown into the tobacco material and also the heated steam having a temperature of 110°C was circulated in a jacket provided at the outer side of the heating container, whereby the tobacco material was heated at 110°C for 30 minutes. After that, the heating container was cooled to room temperature, and the tobacco material was taken out from the container. A nitrous acid concentration and a TSNA concentration in the tobacco material were measured. The results are shown in Table 1.

Example 3

[0021] 5 kg of ground dry oral tobacco material (pH of 8.3) was put in a heating container, and 2% by weight of citric acid and 2% by weight of ascorbic acid were added thereto, thereby adjusting the pH to 5.6. While the pH-adjusted tobacco material was stirred, heated steam having a temperature of 110°C was blown into the tobacco material and also the heated steam having a temperature of 110°C was circulated in a jacket provided at the outer side of the heating container, whereby the tobacco material was heated at 110°C for 30 minutes. After that, the heating container was cooled to room temperature, and the tobacco material was taken out from the container. A nitrous acid concentration and a TSNA concentration in the tobacco material were measured. The results are shown in Table 1.

Comparative Example 1

[0022] 5 kg of ground dry oral tobacco material (pH of 8.3) was put in a heating container, and 2% by weight of citric acid was added thereto, thereby adjusting the pH to 6.5. While the pH-adjusted tobacco material was stirred, heated steam having a temperature of 110°C was blown into the tobacco material and also the heated steam having a temperature of 110°C was circulated in a jacket provided at the outer side of the heating container, whereby the tobacco material was heated at 110°C for 30 minutes. After that, the heating container was cooled to room temperature, and the tobacco material was taken out from the container. A nitrous acid concentration and a TSNA concentration in the tobacco material were measured by the following methods. The results are shown in Table 1.

Comparative Example 2

[0023] 5 kg of ground dry oral tobacco material (pH of 8.3) was put in a heating container. While the tobacco material was stirred, heated steam having a temperature of 110°C was blown into the tobacco material and also the heated steam having a temperature of 110°C was circulated in a jacket provided at the outer side of the heating container, whereby the tobacco material was heated at 110°C for 30 minutes. After that, the heating container was cooled to room temperature, and the tobacco material was taken out from the container. A nitrous acid concentration and a TSNA concentration in the tobacco material were measured. The results are shown in Table 1.

<Measurement Method of TSNA Concentration>

[0024] In accordance with an official method (based on CORESTA METHOD CRM 72), a sample to which an internal standard fluid and 0.1 M ammonium acetate are added is shaken, diluted and filtered, and then the measurement is performed by UPLC/MS/MS.

<Measurement Method of Nitrous Acid Concentration>

[0025] Nitrous acid contained in a sample is reacted with an aromatic primary amine (sulfanilamide) to form a diazonium salt. An azo coupling reaction of the diazonium salt is caused with N-(1-naphthyl)-ehtylenediamine (NED) to form a purplish red azo compound. A concentration of nitrite nitrogen (N) in the sample is measured by measurement of an absorbance (540 nm) of the colored part, and the obtained concentration is converted into a concentration of nitrous acid.

[Table 1]

[0026]

Table 1:

Example No.	pH of tobacco material	Acidic substance	Antioxidant	pH after adjustment	Nitrous acid content after heat treatment (μg/g) (dry basis)	TSNA content after heat treatment (μg/g) (dry basis)
Example 1	8.3	Citric acid	Epigallocatechin gallate	6.1	0.2	2.3
Example 2	8.3	Citric acid	Green tea extract	6.3	0.5	2.5
Example 3	8.3	Citric acid	Ascorbic acid	5.6	1.6	2.0

(continued)

Example No.	pH of tobacco material	Acidic substance	Antioxidant	pH after adjustment	Nitrous acid content after heat treatment (μg/g) (dry basis)	TSNA content after heat treatment (μg/g) (dry basis)
Comparative example 1	8.3	Citric acid	None	6.5	3.8	5.1
Comparative example 2	8.3	None	None	8.3	180.2	3.6

[0027] As is apparent from the results shown in Table 1, according to the present invention, the TSNA content in the tobacco raw material can be remarkably decreased.

Claims

1. A method for producing an oral tobacco material, **characterized by** comprising the steps of:

   adding an antioxidant to a tobacco raw material consisting of a dry oral tobacco material having a pH of 8 or less; and
   heating the antioxidant-added tobacco raw material at a temperature of 80°C or higher.

2. The method according to claim 1, **characterized in that** the tobacco raw material is a material obtained by adding an acidic substance to a tobacco material having a pH of more than 8 to adjust the pH to 8 or less.

3. The method according to claim 2, **characterized in that** the acidic substance is an organic acid.

4. The method according to claim 2, **characterized in that** the acidic substance is a dry oral tobacco material exhibiting acidity.

5. The method according to any one of claims 1 to 4, **characterized in that** the antioxidant is selected from epigallocatechin gallate, ascorbic acid, and a green tea extract.

6. The method according to any one of claims 1 to 5, **characterized in that** the heating is performed at a temperature of 130°C or lower.

7. An oral tobacco material which is obtained by the method according to any one of claims 1 to 6.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2013/054195

A. CLASSIFICATION OF SUBJECT MATTER
A24B13/00(2006.01)i, *A24B15/32*(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, A24B15/32

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	WO 2010/141278 A1 (R.J.REYNOLDS TOBACCO CO.), 09 December 2010 (09.12.2010), page 4, line 8 to page 33, line 3 & JP 2012-528593 A & US 2010/0300463 A1 & US 2011/0048434 A1 & US 2012/0060854 A1 & EP 2437625 A & WO 2012/021683 A2 & CN 102802450 A	1-7
A	WO 2011/093304 A1 (Japan Tobacco Inc.), 04 August 2011 (04.08.2011), paragraph [0040] & US 2012/0298124 A1 & EP 2529634 A1 & TW 201138655 A	1-7

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
17 May, 2013 (17.05.13)	28 May, 2013 (28.05.13)
Name and mailing address of the ISA/ Japanese Patent Office	Authorized officer
Facsimile No.	Telephone No.

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

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2013/054195

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2009-508523 A (R.J. Reynolds Tobacco Co.), 05 March 2009 (05.03.2009), paragraphs [0005] to [0068]; fig. 1 to 5 & JP 5066092 B & JP 2010-534475 A & US 2007/0062549 A1 & US 2007/0186941 A1 & US 2008/0029110 A1 & US 2011/0061666 A1 & EP 1926401 A & EP 2173200 A & EP 2179666 A2 & EP 2377413 A1 & WO 2007/037962 A1 & WO 2009/015142 A2 & CN 101272703 A & CN 101873809 A	1-7

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

REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

- US 2007062549 A **[0004]**