



(11) **EP 1 782 702 A1**

(12) **EUROPEAN PATENT APPLICATION**
published in accordance with Art. 158(3) EPC

(43) Date of publication:
09.05.2007 Bulletin 2007/19

(51) Int Cl.:
A24B 15/12 (2006.01) A24B 3/14 (2006.01)

(21) Application number: **05748851.2**

(86) International application number:
PCT/JP2005/010586

(22) Date of filing: **09.06.2005**

(87) International publication number:
WO 2005/122803 (29.12.2005 Gazette 2005/52)

(84) Designated Contracting States:
AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IS IT LI LT LU MC NL PL PT RO SE SI SK TR

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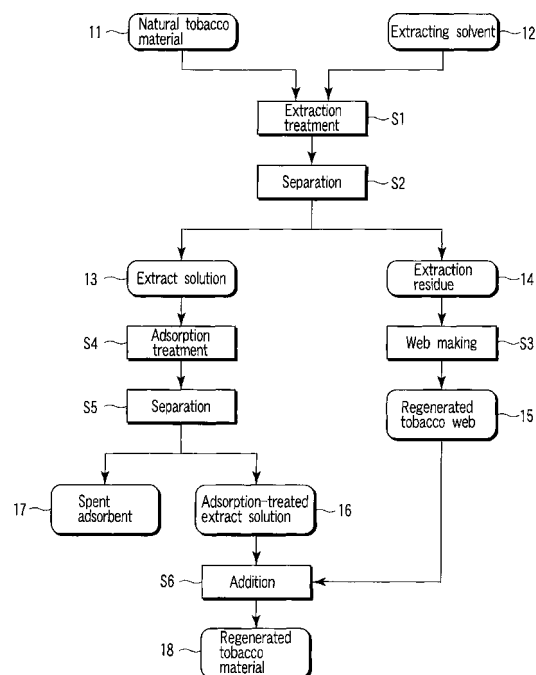
(30) Priority: **16.06.2004 JP 2004178665**

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(54) **PROCESS FOR PRODUCING REGENERATED TOBACCO MATERIAL**

(57) A method of manufacturing a regenerated tobacco material includes extracting a natural tobacco material with an aqueous extracting solvent to obtain an extract solution containing components of the natural tobacco material and an extraction residue, subjecting the extract solution to an adsorption separation operation using an adsorbent to reduce an amount of nitrosamines, preparing a regenerated tobacco web using the extraction residue, and adding at least a part of the extract solution in which the amount of nitrosamines is reduced, to the regenerated tobacco web. The adsorbent is a strongly hydrophobic adsorbent. The adsorption separation operation is carried out within 60 minutes using the strongly hydrophobic adsorbent in an amount corresponding to 10 to 110% of a weight of soluble components of the extract solution.



FIGURE

Description

Technical Field

5 **[0001]** The present invention relates to a method of manufacturing a regenerated tobacco material.

Background Art

10 **[0002]** Tobacco materials such as natural tobacco leaves, cut tobacco, midribs, stems and roots contain various components including nicotine, nitrosamines, hydrocarbons and proteins. In the process of manufacturing regenerated tobacco materials, these components, extracted from a natural tobacco material, are put back to a regenerated tobacco web manufactured from a residue of the extraction. Some of these natural tobacco material components should desirably be reduced in amount or removed, while other ones should not be removed or even increased in amount, for a reason of smoking flavor or some other reason.

15 **[0003]** For example, International Publication WO 02/28209 discloses a method in which an extract solution obtained by extracting tobacco with a solvent is brought into contact with an adsorbent such as activated carbon or zeolite in order to reduce tobacco-specific nitrosamines (TSNAs). This patent document 1 further discloses that an extract solution reduced in nitrosamines is added to a regenerated tobacco material.

20 **[0004]** For example, U.S. Patent 5,810,020 discloses that nitrosamines are removed by a liquid-liquid extraction treatment using a great amount of crown ether.

[0005] International Publication WO 01/65954 discloses that tobacco is treated with supercritical carbon dioxide to extract nitrosamines and the extract is subjected to a nitrosamine removing process.

25 **[0006]** Nitrosamines, TSNAs in particular, contained in natural tobacco materials are substances which should desirably be eliminated. However, TSNAs are similar in structure to nicotine, which is a useful component, and therefore it is very difficult to remove TSNAs selectively without removing nicotine. With the conventional technique, it is therefore difficult to prepare a regenerated tobacco material which contains a significant amount of nicotine, but is substantially freed of TSNAs, by using the extract solution obtained from the natural tobacco material and a fibrous component, which is an extraction residue.

30 **[0007]** More specifically, with the method of International Publication WO 02/28209, it is possible to separate TSNAs from nitric acid or saccharides, but nicotine is adsorbed and removed together with TSNAs by the adsorbent.

[0008] Further, the method of U.S. Patent No. 5810020 is not desirable in terms of safety since it uses a great amount of crown ether. Further, the method of International Publication WO 01/65954 involves a supercritical extraction process which is costly, and therefore its practical utility is not high.

35 Disclosure of Invention

[0009] Therefore, it is an object of the present invention to provide a method of manufacturing a regenerated tobacco material that contains a significant amount of nicotine but substantially freed of TSNAs, by separating nicotine and TSNAs in an extract solution obtained from a natural tobacco material in a relatively simple manner.

40 **[0010]** The present invention provides a method of manufacturing a regenerated tobacco material using an extract solution obtained by extraction from a natural tobacco material with an aqueous extracting solvent, and an extraction residue.

45 **[0011]** The inventors of the present invention pay attention to a highly hydrophobic adsorbent such as activated carbon as simple means for separating nicotine and TSNAs in an extract solution obtained from a natural tobacco material by extraction with an aqueous extracting solvent. A strongly hydrophobic adsorbent such as activated carbon has a tendency of adsorbing TSNAs together with nicotine. However, the inventors of the present invention has found that TSNAs can be significantly removed while suppressing the amount of adsorption of nicotine to a minimum level by controlling the adsorption time and the amount of the adsorbent employed. The present invention is based on these findings.

50 **[0012]** Thus, according to the present invention, there is provided a method of manufacturing a regenerated tobacco material, comprising (a) extracting a natural tobacco material with an aqueous extracting solvent to obtain an extract solution containing components of the natural tobacco material, and an extraction residue, (b) subjecting the extract solution to an adsorption separation operation using an adsorbent to reduce an amount of nitrosamines, (c) preparing a regenerated tobacco web using the extraction residue, and (d) adding at least a part of the extract solution, in which the amount of nitrosamines has been reduced, to the regenerated tobacco web, wherein the adsorbent is a strongly hydrophobic adsorbent, and the adsorption separation operation is carried out within 60 minutes, using the strongly hydrophobic adsorbent in an amount corresponding to 10 to 110% of the weight of soluble components in the extract solution.

Brief Description of Drawings

[0013] The single figure is a flow diagram illustrating a method of manufacturing a regenerated tobacco material according to an embodiment of the present invention.

Best Mode for Carrying Out the Invention

[0014] The present invention will be described below in more detail with reference to various embodiments.

[0015] The present invention is a method of manufacturing a regenerated tobacco material by substantially removing TSNA's (N'-nitrosonornicotine (NNN), 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone (NNK), N'-nitrosoanatabine (NAT), etc.) from an extract solution obtained by extracting a natural tobacco material with an aqueous extracting solvent while suppressing loss of nicotine as much as possible, and adding the extract solution to a regenerated tobacco web prepared by using the extraction residue.

[0016] FIG. 1 is a flow diagram illustrating a method of manufacturing a regenerated tobacco material according to an embodiment of the present invention.

[0017] As shown in FIG. 1, first, a natural tobacco material 11 is subjected to an extraction treatment S1 by mixing and stirring the natural tobacco material 11 and an extracting solvent 12.

[0018] As the natural tobacco material 11, tobacco leaves, cut tobacco, midribs, stems, roots and a mixture of these can be used. As the extracting solvent, an aqueous solvent can be used. The aqueous extracting solvent such as water may be alkaline or acidic. It is also possible to use, as the aqueous extracting solvent, a mixture of water and a water-miscible organic solvent as well. Examples of such an organic solvent are alcohols such as ethanol. An inorganic salt such as sodium hydroxide may be dissolved in these extracting solvents. The extraction treatment S1 is carried out usually at a temperature of 50 to 100°C for 5 minutes to 6 hours.

[0019] After finishing the extraction treatment S1, the extraction mixture obtained is subjected to a separation operation S2 such as filtration to separate it into an extract solution 13 and an extraction residue 14. The extract solution 13 contains watersoluble components in the natural tobacco material, such as nicotine and TSNA's.

[0020] The extraction residue 14 consists essentially of fibers. A regenerated tobacco web 15 is manufactured by an ordinary method, using the extraction residue 14. The regenerated tobacco web 15 may be partially or entirely constituted by the extraction residue 14.

[0021] On the other hand, the extract solution 13 obtained from the separation operation S2 is subjected to an adsorption treatment operation S4 using a strongly hydrophobic adsorbent. In the present invention, the adsorption separation operation S4 can be carried out by bringing the extract solution 13 into contact with the strongly hydrophobic adsorbent. The strongly hydrophobic adsorbent is an adsorbent generally known to have a high hydrophobicity. Examples thereof are activated carbon, a styrene-based porous resin, and an adsorbent in which a modifying group that further enhances the hydrophobic property (hydrophobic group), for example, bromine, is introduced to a styrene-based porous resin. With use of the strongly hydrophobic adsorbent, TSNA's can be significantly eliminated while suppressing the loss of nicotine from the tobacco extract.

[0022] The contact between the extract solution 13 and the strongly hydrophobic adsorbent can be achieved by adding particles of the strongly hydrophobic adsorbent to the extract 13, followed by stirring. Alternatively, the contact between the extract 13 and the strongly hydrophobic adsorbent can be achieved by passing the extract solution 13 through an adsorbent bed filled with particles of the strongly hydrophobic adsorbent.

[0023] The amount of the strongly hydrophobic adsorbent used is that corresponds to 10 to 110% of the weight of the soluble components in the extract solution 13. If the amount of the strongly hydrophobic adsorbent is less than 10%, TSNA's in the extract solution cannot be sufficiently removed. On the other hand, if the amount of the adsorbent exceeds 110%, the amount of nicotine adsorbed significantly increases. It is preferable that the amount of the strongly hydrophobic adsorbent correspond to 10 to 50% of the weight of the soluble components in the extract 13. It should be noted that the amount of the soluble components in the extract can be determined by the freeze-dry method.

[0024] The contact time between the extract solution 13 and the strongly hydrophobic adsorbent is within 60 minutes. When the contact time is within 60 minutes, TSNA's can be eliminated while significantly suppressing the adsorption of nicotine. The contact time is preferably 5 to 60 minutes, and more preferably, 5 to 30 minutes.

[0025] In order to suppress the adsorption of nicotine and enhance the adsorption of TSNA's, the pH value of the extract when brought into contact with the strongly hydrophobic adsorbent is preferably more than 2 but less than 10, and more preferably, more than 4 but less than 10 in the present invention. The pH value can be adjusted by adding acid or alkali. Further, the temperature of the extract solution is preferably 0 to 100°C, and more preferably 20 to 60°C.

[0026] The extract that has been subjected to adsorption/separation is separated (S5) from the strongly hydrophobic adsorbent (spent adsorbent 17). Where the contact between the extract and the strongly hydrophobic adsorbent are carried out by adding the adsorbent to the extract and stirring, this separation can be carried out by filtration. On the other hand, when the contact between the extract and the strongly hydrophobic adsorbent is carried out by passing the

extract through the strongly hydrophobic adsorbent bed, the extract after the adsorption process is, needless to mention, is flowed out of the bed of the strongly hydrophobic adsorbent in the state separated from the adsorbent. The thus obtained extract 16 already subjected to the adsorption treatment is added (S5) partially or entirely to the regenerated tobacco web 15 after concentrating or without concentrating the extract, and thus a regenerated tobacco material 18 can be obtained. A cigarette manufactured using the regenerated tobacco material 18 thus obtained exhibits a significantly reduced amount of TSNAs in the smoke when it is burned.

[0027] The above description is provided as embodiments of the present invention, and the invention is not limited to these embodiments. Needless to say, the above various embodiments can be combined together.

[0028] The present invention will now be described with reference to various Examples, but the invention is not limited to these Examples either.

[0029] In the following Examples, the amount of TSNAs was measured according to the method of measuring TSNAs in the cut tobacco of Arista (Official Method T-309, Determination of Nitrosamines in Whole Tobacco, made by the Department of Health, dated December 31, 1999). More specifically, for the measurement, the tobacco extract or adsorbent-treated liquid was subjected to LC-MS/MS. The amount of nicotine was measured by a method in compliance with German Institute for Normalization DIN 10373. More specifically, sodium hydroxide was added to the tobacco extract or adsorbent-treated liquid to make it alkaline, and then hexane was further added thereto. The resultant was mixed to make nicotine to transfer to the hexane phase, and the hexane phase alone was subjected to GC/FID for measurement.

Examples 1 - 8

[0030] At a temperature of 25°C, 100g of cut tobacco leaves (X-KL1) was mixed with 1000 mL of water and the mixture was stirred, thereby performing the extraction of the cut tobacco. Thus obtained extraction mixture was filtrated to separate it into an extract solution and an extraction residue. A regenerated tobacco web was made from the extraction residue.

[0031] On the other hand, activated carbon (average particle diameter: 1100 μm ; specific surface area: 1638 m^2/g) was added to the extract solution (pH: 6.0) in amounts (%) indicated in TABLE 1 with respect to the amount of soluble components of the extract solution, stirred for time periods (adsorption treatment times) indicated in TABLE 1 at a temperature of 25°C, and then filtrated. The total amount of TSNAs and amount of nicotine contained in the extract solution (filtrate) after the adsorption treatment were analyzed. The results are also indicated in TABLE 1. TABLE 1 further indicates the rate of change in total TSNA and nicotine. In the TABLE 1, "untreated" indicates the extract that was not subjected to the adsorption treatment (The same applies to TABLES 2 to 6 below).

TABLE 1

	Amount of adsorbent (%)	Adsorption treatment time (min)	Total amount of TSNAs ($\mu\text{g/g}$)	Amount of nicotine (mg/g)	Rate of change in total TSNAs (a)	Rate of change in nicotine (b)	Difference (a-b)
Untreated	-	-	30.5	23.4	-	-	-
Ex. 1	13	15	11.8	17.2	-61%	-26%	-35%
Ex. 2	13	30	10.0	16.4	-67%	-30%	-37%
Ex. 3	13	60	8.9	14.1	-71%	-39%	-31%
Ex. 4	13	120	8.5	14.0	-72%	-40%	-32%
Ex. 5	25	15	7.7	21.3	-75%	-9%	-66%
Ex. 6	25	30	4.1	17.6	-86%	-25%	-62%
Ex. 7	25	60	2.6	15.0	-91%	-36%	-56%
Ex. 8	50	5	6.2	15.5	-80%	-34%	-46%

[0032] From the results shown in TABLE 1, it can be seen that when the adsorbent (activated carbon) was used in an amount corresponding to 10 to 50% of the amount of the soluble components in the extract and the adsorption treatment was carried out for 5 to 60 minutes, TSNAs were decreased by 60% or more while suppressing the decrease rate of the amount of nicotine within less than 40% (Examples 1 - 3 and 5 - 8). In particular, when the amount of the adsorbent employed was equivalent to 25% of that of the soluble components in the extract and the adsorption treatment was carried out for 15 to 30 minutes, the difference between the rate of change in the total TSNA and the rate of change

in nicotine was as much as -62 to -66%, and indicating that the TSNA-selective removability becomes high (Examples 5 - 6.)

[0033] The extracts subjected to the adsorption treatment in Examples 1 - 3 and 5 - 6 were added to the regenerated tobacco webs, respectively, preparing regenerated tobacco materials.

Examples 9 to 11

[0034] The same operations were carried out as in Examples 1 - 8 except that a bromine chemically bonded styrene resin-based synthetic adsorbent (SP 207 available from Mitsubishi Chemical Corporation) was used as the strongly hydrophobic adsorbent in respective amounts indicated in TABLE 2, and stirred together with the extract for respective time periods indicated in TABLE 2. The results of the analysis of the total amount of TSNA and amount of nicotine contained in the extract (filtrate) after the adsorption treatment were also indicated in TABLE 2.

TABLE 2

	Amount of adsorbent (%)	Adsorption treatment time (min)	Total amount of TSNA (μg/g)	Amount of nicotine (mg/g)	Rate of change in total TSNA (a)	Rate of change in nicotine (b)	Difference (a-b)
Untreated	-	-	32.3	20.4	-	-	-
Ex. 9	50	15	8.7	18.9	-73%	-7%	-66%
Ex. 10	50	30	7.2	18.1	-78%	-11%	-66%
Ex. 11	50	60	5.9	15.9	-82%	-22%	-60%

[0035] From the results shown in TABLE 2, it can be seen that the total amount of TSNA was decreased by 70% or more while suppressing the decrease rate of the amount of nicotine within 25% in any of the Examples. In particular, when the amount of the adsorbent employed was equivalent to 50% of that of the soluble components in the extract and the adsorption treatment was carried out for 15 to 30 minutes, the difference between the rate of change in the total TSNA and the rate of change in nicotine was much as -66%, and indicating that the TSNA-selective removability becomes high (Examples 9 - 10.)

[0036] The extracts subjected to the adsorption treatment in Examples 9 - 11 were added to the regenerated tobacco webs, respectively, preparing regenerated tobacco materials.

Examples 12 - 14

[0037] The same operations were carried out as in Examples 1 - 8 except that activated carbon having an average particle diameter of 150 μm and a specific surface area of 1140 m²/g was used as the strongly hydrophobic adsorbent in respective amounts indicated in TABLE 3, and stirred together with the extract for respective time periods indicated in TABLE 3. The results of the analysis of the total amount of TSNA and amount of nicotine contained in the extract (filtrate) after the adsorption treatment were also indicated in TABLE 3.

TABLE 3

	Amount of adsorbent (%)	Adsorption treatment time (min)	Total amount of TSNA (μg/g)	Amount of nicotine (mg/g)	Rate of change in total TSNA (a)	Rate of change in nicotine (b)	Difference (a-b)
Untreated	-	-	30.5	23.4	-	-	-
Ex. 12	50	15	3.9	22.6	-87%	-3%	-84%
Ex. 13	50	30	2.8	19.3	-91%	-17%	-73%
Ex. 14	50	60	1.9	15.3	-94%	-35%	-59%

[0038] From the results shown in TABLE 3, it can be seen that the total amount of TSNA was decreased by 70% or more while suppressing the decrease rate of the amount of nicotine within 35% in any of the Examples. In particular,

when the amount of the adsorbent employed was equivalent to 50% of that of the soluble components in the extract and the adsorption treatment was carried out for 15 to 30 minutes, the difference between the rate of change in the total TSNAs and the rate of change in nicotine was much as -73 to -84%, indicating that the TSNA-selective removability becomes high (Examples 12 - 13).

[0039] The extracts subjected to the adsorption treatment in Examples 12 - 14 were added to the regenerated tobacco webs, respectively, preparing regenerated tobacco materials.

Examples 15 to 20

[0040] The cut tobacco was extracted in accordance with the procedures of Examples 1 - 8, and the extracts thus obtained were adjusted to a pH of 2.0 or 10.0 by adding acid (acetic acid) or alkali (sodium hydroxide). The extracts were treated with activated carbon used in Examples 12 - 14 under respective conditions indicated in TABLE 4. The results of the analysis of the total amount of TSNAs and amount of nicotine contained in the extract (filtrate) after the adsorption treatment were also indicated in TABLE 4.

TABLE 4

	pH of extract solution	Amount of adsorbent (%)	Adsorption treatment time (min)	Total amount of TSNAs ($\mu\text{g/g}$)	Amount of nicotine (mg/g)	Rate of change in total TSNAs (a)	Rate of change in nicotine (b)	Difference (a-b)
Untreated		-	-	30.5	23.4	-	-	-
Ex. 15	2.0	50	15	23.8	23.3	-22%	0%	-22%
Ex. 16	2.0	50	30	23.4	22.9	-23%	-2%	-21%
Ex. 17	2.0	50	60	24.1	22.5	-21%	-4%	-17%
Ex. 18	10.0	50	15	1.3	3.0	-96%	-87%	-9%
Ex. 19	10.0	50	30	0.8	1.5	-97%	-93%	-4%
Ex. 20	10.0	50	60	0.9	1.1	-97%	-95%	-2%

[0041] As can be seen from TABLE 4, when the pH value of the extract was 2.0, both of TSNAs and nicotine were not easily adsorbed (Examples 15 - 17), whereas when the pH value of the extract is 10.0, about 90% or more of both of TSNAs and nicotine were removed (Examples 18 - 20). In consideration of the cases of Examples 12 to 14, in which the pH value of the extract was 6.0 and the TSNA-selective adsorption property was excellent, it can be seen that the pH value of the extract is preferably more than 2 but less than 10. The results were considered to be due to the fact that the pKa of TSNAs was 4 and pKa of nicotine was 10. Thus, it is preferable for a higher TSNA-selective removal by adsorption that the pH value of the extract be higher than 4.0 where TSNAs are ionized, but lower than 10 where nicotine is ionized.

Examples 21 - 38

[0042] The same procedures were followed as in Examples 1 - 8 except that activated carbon was used under different conditions as indicated in TABLE 5. The results of the analysis of the total amount of TSNAs and amount of nicotine contained in the extract (filtrate) after the adsorption treatment were also indicated in TABLE 5.

TABLE 5

	Amount of adsorbent (%)	Adsorption treatment time (min)	Total amount of TSNAs ($\mu\text{g/g}$)	Amount of nicotine (mg/g)	Rate of change in total TSNAs (a)	Rate of change in nicotine (b)	Difference (a-b)
Untreated	-	-	30.5	23.4	-	-	-
Ex. 21	2	15	26.2	20.4	-14%	-13%	-2%
Ex. 22	2	30	27.9	19.6	-9%	-16%	7%

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(continued)

	Amount of adsorbent (%)	Adsorption treatment time (min)	Total amount of TSNA's ($\mu\text{g/g}$)	Amount of nicotine (mg/g)	Rate of change in total TSNA's (a)	Rate of change in nicotine (b)	Difference (a-b)
Ex. 23	2	60	27.7	19.3	-9%	-18%	8%
Ex. 24	2	120	24.8	19.1	-19%	-18%	0%
Ex. 25	5	15	27.4	18.2	-10%	-22%	12%
Ex. 26	5	30	24.5	18.1	-20%	-22%	3%
Ex. 27	5	60	22.3	16.2	-27%	-31%	4%
Ex. 28	5	120	16.6	16.1	-45%	-31%	-14%
Ex. 29	50	120	3.7	3.3	-88%	-86%	-2%
Ex. 30	125	30	2.2	0.2	-93%	-99%	7%
Ex. 31	250	5	0.1	4.2	-100%	-82%	-18%
Ex. 32	250	15	1.8	1.7	-94%	-93%	-1%
Ex. 33	250	30	1.1	0.6	-96%	-98%	1%
Ex. 34	250	60	1.0	0.7	-97%	-97%	0%
Ex. 35	500	5	0.0	1.4	-100%	-94%	-6%
Ex. 36	500	15	0.4	0.5	-99%	-98%	-1%
Ex. 37	500	30	0.0	0.0	-100%	-100%	0%
Ex. 38	500	60	0.0	0.0	-100%	-100%	0%

[0043] As can be seen from the results shown in TABLE 5, when the amount of the adsorbent used and the adsorption time period fall out of the ranges specified by the present invention, it is not possible to achieve the advantageous effect of enhancing the adsorption of TSNA's while suppressing the adsorption of nicotine.

Examples 39 - 47

[0044] The same operations were carried out as in Examples 1 to 8 except that active alumina, which is a hydrophilic adsorbent, was used as the adsorbent in respective amounts indicated in TABLE 6, and stirred together with the extract for a respective time period indicated in TABLE 6. The results of the analysis of the total amount of TSNA's and amount of nicotine contained in the extract (filtrate) after the adsorption treatment were also indicated in TABLE 6.

TABLE 6

	Amount of adsorbent (%)	Adsorption treatment time (min)	Total amount of TSNA's ($\mu\text{g/g}$)	Amount of nicotine (mg/g)	Rate of change in total TSNA's (a)	Rate of change in nicotine (b)	Difference (a-b)
Untreated	-	-	33.7	25.1	-	-	-
Ex. 39	50	15	30.7	22.9	-9%	-9%	0%
Ex. 40	50	30	30.7	23.5	-9%	-6%	-3%
Ex. 41	50	60	33.3	24.4	-1%	-3%	2%
Ex. 42	250	15	31.8	23.2	-6%	-8%	2%
Ex. 43	250	30	32.8	22.8	-3%	-9%	6%
Ex. 44	250	60	32.6	23.7	-3%	-6%	2%

(continued)

	Amount of adsorbent (%)	Adsorption treatment time (min)	Total amount of TSNAs ($\mu\text{g/g}$)	Amount of nicotine (mg/g)	Rate of change in total TSNAs (a)	Rate of change in nicotine (b)	Difference (a-b)
Ex. 45	500	15	32.2	24.4	-5%	-3%	-2%
Ex. 46	500	30	33.3	24.7	-1%	-1%	0%
Ex. 47	500	60	33.1	25.0	-2%	0%	-2%

[0045] As can be seen from TABLE 6, when the hydrophilic adsorbent is used, TSNAs are not substantially eliminated.

[0046] As described above, according to the present invention, it is possible to separate nicotine and TSNAs from an extract solution obtained from a natural tobacco material in a relatively simple manner, and to manufacture a regenerated tobacco material substantially freed of TSNAs while suppressing the loss of nicotine as much as possible.

Claims

1. A method of manufacturing a regenerated tobacco material, comprising:

- (a) extracting a natural tobacco material with an aqueous extracting solvent to obtain an extract solution containing components of the natural tobacco material and an extraction residue;
- (b) subjecting the extract solution to an adsorption separation operation using an adsorbent to reduce an amount of nitrosamines;
- (c) preparing a regenerated tobacco web using the extraction residue; and
- (d) adding at least a part of the extract solution in which the amount of nitrosamines has been reduced, to the regenerated tobacco web,

wherein the adsorbent is a strongly hydrophobic adsorbent, and the adsorption separation operation is carried out within 60 minutes using the strongly hydrophobic adsorbent in an amount corresponding to 10 to 110% of a weight of soluble components of the extract solution.

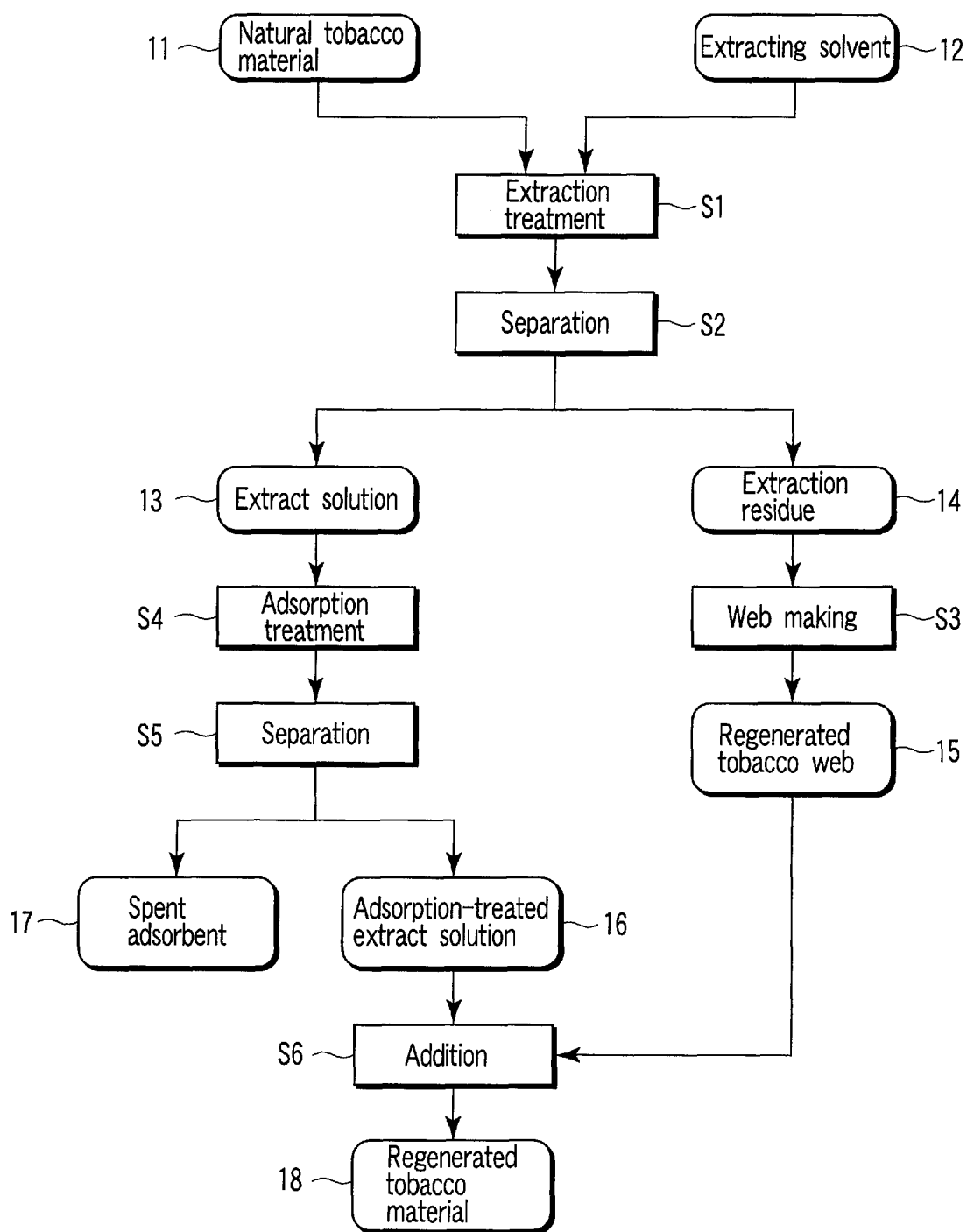
2. The method according to claim 1, wherein the adsorbent is a styrene-based porous resin, a activated carbon or an adsorbent in which a hydrophobic group is bonded to a surface of the adsorbent.

3. The method according to claim 1, wherein the strongly hydrophobic adsorbent is used in an amount corresponding to 15 to 60% of a weight of the soluble components of the extract solution.

4. The method according to claim 1, wherein a pH value of the extract solution is more than 2 but less than 10.

5. The method according to claim 1, wherein a pH value of the extract is more than 4 but less than 10.

6. The method according to claim 1, wherein the adsorption separation operation is carried out at a temperature of 0 to 100°C.



FIGURE

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2005/010586

A. CLASSIFICATION OF SUBJECT MATTER
Int.Cl.⁷ A24B15/12, A24B3/14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Int.Cl.⁷ A24B15/12, A24B3/14, 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 1996-2005
Kokai Jitsuyo Shinan Koho 1971-2005 Toroku Jitsuyo Shinan Koho 1994-2005

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2004-510422 A (BASKEVITCH, Nicolas; LEBEC, lanig; RAVERDY-LAMBERT, Diane), 08 April, 2004 (08.04.04), & US 2002/134394 A1 & EP 1322191 A & WO 02/28209 A1 & AU 9658001 A & BR 114448 A & CN 1477935 A	1-6
A	US 2004/0045565 A1 (BROWN & WILLIAMSON TOBACCO CORP.), 11 March, 2004 (11.03.04), & WO 2004/21809 A1 & CA 2502674 A	1-6

☒ 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
29 August, 2005 (29.08.05)

Date of mailing of the international search report
20 September, 2005 (20.09.05)

Name and mailing address of the ISA/
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INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2005/010586

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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REFERENCES CITED IN THE DESCRIPTION

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