



(11) **EP 3 263 276 A1**

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

(43) Date of publication:
03.01.2018 Bulletin 2018/01

(51) Int Cl.:
B24B 15/00 (2006.01)

(21) Application number: **16876978.4**

(86) International application number:
PCT/CN2016/108256

(22) Date of filing: **01.12.2016**

(87) International publication number:
WO 2017/181685 (26.10.2017 Gazette 2017/43)

(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
Designated Validation States:
MA MD

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(30) Priority: **20.04.2016 CN 201610247577**

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(54) **METHOD FOR MANUFACTURING HEAT-NOT-BURN TOBACCO SUBSTRATE CONTAINING ACTIVATED CARBON**

(57) The present invention discloses a method for preparing heat-not-burn tobacco substrate containing activated carbon, which comprises the following steps: activated carbon is weighed, mixed with water, deflaked and pulped, to obtain an activated carbon slurry; tobacco raw material is weighed and subjected to soaking, filtering and pulping to obtain a tobacco slurry; by weight parts, 50~80 parts of tobacco slurry, 8~25 parts of wood pulp, 10~30 parts of activated carbon slurry and 4~8 parts of binder are weighted and mixed well; the mixed material

is shaped and dried to obtain a tobacco substrate; by weight parts, 1~40 parts of tobacco essential oil, 2~20 parts of tobacco flavor and 50~80 parts of atomizing agent are weighted and mixed well to obtain a coating liquid; the coating liquid is sprayed onto the tobacco substrate, and allowed to stand for 40 to 48 hours under constant temperature and humidity conditions to finally obtain a heat-not-burn tobacco substrate containing activated carbon.

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Description

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims priority to Chinese patent application No. 201610247577.1, titled "METHOD FOR PREPARING HEAT-NOT-BURN TOBACCO SUBSTRATE CONTAINING ACTIVATED CARBON", filed with the Chinese State Intellectual Property Office on April 20, 2016, the entire contents of which are incorporated herein by reference.

FIELD

[0002] The present disclosure relates to the field of tobacco technology, in particular to a method for preparing heat-not-burn tobacco substrate containing activated carbon.

BACKGROUND

[0003] Heat-not-burn cigarette is to provide consumers with nicotine and tobacco-flavored aromas by heating non-combustible tobacco to meet consumer demand while significantly reducing the release of tar and harmful substances from mainstream smoke gases. It is an important development trend in the future tobacco market. The design of non-combustible tobacco raw materials is very different from traditional cigarettes. Relevant experiments have shown that ordinary cigarettes that have not been specially treated can not provide satisfactory amount of smog and aroma when used in general cigarette utensils.

[0004] Patent document (publication No. CN 103181613) provides a method for the preparation of an electrically dry distilled tobaccosheets. The patent document discloses that pasting tobacco extracts, plant extracts and smoke producing agent to tobacco sheets can be used to produce smoke in electrical distillers. However, the patent document simply modifies the ordinary sheets in a sheet-coating manner, and those tobacco sheets without special treatment have a low adsorption amount and lack of tobacco aroma during use, particularly when coating amount is increased, its performance will be greatly reduced.

SUMMARY

I. Technical Problem to be Solved

[0005] The technical problem to be solved by the present disclosure is how to provide a heat-not-burn tobacco substrate having a nicotine and tobacco characteristic aroma while substantially reducing the amount of tar and harmful substances in the smoke gases, i.e., providing a method for preparing heat-not-burn tobacco substrate containing activated carbon.

II. Technical Solution

[0006] In order to solve the above technical problem, the present disclosure provides a method for preparing heat-not-burn tobacco substrate containing activated carbon, which comprises the following steps (all materials are commercially available):

Step 1: Preparation of activated carbon slurry

[0007] Activated carbon is weighed, mixed with water, deflaked and pulped, to make an activated carbon slurry with a concentration of 1~5%.

Step 2: Preparation of tobacco slurry

[0008] Tobacco raw material is weighed and put into water for soaking. Filtration is carried out after soaking, and the filtered tobacco is mechanically pulped to obtain a tobacco slurry.

Step 3: Preparation of tobacco substrates

[0009] By weight parts, 50~80 parts of tobacco slurry, 8~25 parts of wood pulp, 10~30 parts of activated carbon slurry and 4~8 parts of binder are weighted and mixed well. The mixed material is fed into a paper machine to form a wet tobacco substrate and dried by hot air to obtain a tobacco substrate.

Step 4: Preparation of Coating Liquid

[0010] By weight parts, 1~40 parts of tobacco essential oil, 2~20 parts of tobacco flavor and 50~80 parts of atomizing agent are weighted and mixed well to obtain a coating liquid.

Step 5: Coating Tobacco Substrates

[0011] The coating liquid obtained in Step 4 is weighed according to the weight of 25~40% of the tobacco substrate obtained in Step 3, and sprayed onto the tobacco substrate. The tobacco substrate is allowed to stand for 40~48 hours under constant temperature and humidity conditions to obtain a heat-not-burn tobacco substrate containing activated carbon.

[0012] Preferably, in Step 1, the activated carbon particles are activated carbon particles and/or activated carbon fibers; the activated carbon particles have a particle size of 80~100 meshes; and the activated carbon fibers have a fiber length of 0.5~8mm, preferably 0.5~5mm, more preferably 1mm.

[0013] The above-mentioned activated carbon is preferably a wooden activated carbon or a fruit-shell activated carbon.

[0014] Pretreatment method of the activated carbon particles: 40~200 meshes plant source activated carbon is added to 60~100°C hot water, sonicated under a fre-

quency of 20~60KHz for 30~60min, followed by filtration, washing and drying in a high temperature of 100~200°C with ventilation.

[0015] Pretreatment method of the activated carbon fibers: activated carbon fibers are added to hot water and boiled for 30~60 min, followed by filtration, washing and drying at a high temperature of 100~200°C with ventilation.

[0016] Preferably, in Step 2, the tobacco material comprises one or more of tobacco stem, tobacco powder, crumbled tobacco leaf and tobacco shred.

[0017] Preferably, in Step 3, the wood pulp is one or more of spruce bleached kraft pulp, poplar bleached kraft pulp, needle leave wood bleached kraft pulp and eucalyptus bleached kraft pulp; the binder is one or more of xanthate gum, arabic gum and guar gum.

[0018] Preferably, in Step 3, the obtained tobacco substrate has a moisture content of 10~15%, a thickness of 10~300 μ m and a thickness/weight (g) of 20~200 g/m².

[0019] Preferably, in Step 4, the tobacco essential oil is prepared by the following method:

[0020] Tobacco leaf or tobacco shred is pulverized by a pulverizer and passed through a 40~100 mesh sieve; a certain ratio of solvent water, ethanol or ethyl ether is added for ultrasonic extraction at a certain temperature for 2~10h. After sedimentation, filtration and concentration under reduced pressure, tobacco primary extract is obtained and subjected to molecular distillation for fine extraction to obtain tobacco essential oil. Conditions for the molecular distillation are as following: feed rate 2~10mL/min, vacuum degree 15~20Pa, injection temperature 80°C, heating temperature 60~80°C, cooling temperature 10~20°C, rotation speed 300~400r/min. The tobacco leaf or tobacco shred is one or more of flue-cured tobacco, aired-cured tobacco or sun-cured tobacco; the aired-cured tobacco is selected from one or more of beuley tobacco or Maryland tobacco; the sun-cured tobacco is selected from one or more of aromatic tobacco, sun-cured red tobacco, sun-cured yellow tobacco or rustica tobacco.

[0021] Preferably, in Step 4, the tobacco flavor is one or more of vanillin, benzoic acid, phenylacetic acid, 10% Peru extractum, 5% tree moss extractum, 90% clove oil, anisaldehyde, 10% pineapple ketone, benzyl alcohol, phenylethyl alcohol, acetyltoylene, ethyl phenylacetate, plum extractum, phenylacetaldehyde, acetophenone, angelica lactone, farnesyl ketone, dodecanoic acid, isovaleric acid, benzyl butyrate, octanol, tetramethylpyrazine, citric acid, ethyl maltol, sclareol, sclareolide, ambergris ether, davana oil, orange leaf oil, valerian oil, vetiver oil, labdanum extractum, storax extractum, angelica root tincture, extractum tamarind liquidum, tamarind extractum, chamomile extractum, vanilla extractum, fig extractum, apple juice, megastigmatrienone, dihydrokawifruitlactone, β -ionone, palmitic acid and geranyl acetone. By mass percentage, the following combination is preferred: vanillin 1%, benzoic acid 0.2%, phenylacetic acid 0.5%, Peru extractum 1.0%, tree moss extractum

5%, clove oil 0.5%, anisaldehyde 0.5%, pineapple aldehyde 0.5%, phenylethanol 0.4%, acetyltoylene 0.2%, ethyl phenylacetate 0.2%, ethanol 25% and propylene glycol 65%.

[0022] Preferably, in Step 4, the atomizing agent is one or more of propylene glycol, triethylene glycol, 1,3-butanediol, glycerol, dipropylene glycol and polyethylene glycol; glycerol is preferred.

[0023] The present disclosure also provides a tobacco substrate prepared by the above method for preparing heat-not-burn tobacco substrate containing activated carbon.

[0024] The present disclosure also provides use of the tobacco substrate prepared by the method for preparing heat-not-burn tobacco substrate containing activated carbon in heating non-combustible cigarette utensils, i.e., the tobacco substrate is rolled and made into cigarettes, and placed in the cigarette utensils. The heating temperature is set to 200~500°C and the heating time is 10~30s. After the heating time is reached, the cigarette is ready for smoking.

III. Beneficial Effect

[0025] In the method of the present disclosure, adding activated carbon to the substrate can increase the content of atomizing agent in the substrate, thus significantly improving the amount of smog in the smoking process when using a heating non-combustible cigarette utensil. Adding activated carbon to the substrate can also improve the ability of the substrate to load tobacco flavor and tobacco essential oil, significantly increasing the amount of aroma and tobacco fragrance when using a heating non-combustible cigarette utensil. Pretreatment of activated carbon can minimize the effect of activated carbon on tobacco aroma.

DETAILED DESCRIPTION OF EMBODIMENTS

[0026] The embodiments of the present disclosure will now be described in further detail with reference to the following examples. The following examples are merely illustrative of the present disclosure and are not intended to limit the scope of the invention.

Example 1

[0027] Coconut shell activated carbon of 80~100 meshes was added to 80°C hot water, sonicated under a frequency of 40KHz for 60min, followed by filtration, washing and drying at a high temperature of 150°C with ventilation.

[0028] By mass percentage, tobacco pulp 65.5%, wood pulp 14.5%, coconut shell activated carbon slurry 15% and xanthate gum 5% were mixed well and fed directly into a twin-wire papermachine for shaping. The tobacco substrate was obtained by drying with a hot-air dryer, and the resulting tobacco substrate had a thick-

ness of 100 μ m, and a thickness weight (g) of 50 g/m².

[0029] Coating liquid comprising, by mass percentage, flue-cured tobacco essential oil 20%, tobacco flavor 6%, glycerol 74% was prepared. 25% of the coating liquid based on the mass percentage of the tobacco substrate was sprayed onto the tobacco substrate, and balanced in a constant temperature and humidity incubator at an ambient temperature (22 $\pm$ 1) °C and relative humidity (60 $\pm$ 2)% for 48h to obtain tobacco substrate sample 1#.

[0030] The above substrate was rolled and made into a tobacco roll, compounded with a hollow filter rod to form a cigarette and inserted into a cigarette utensil for use. The heating temperature for the cigarette utensil was set to 250°C, and smoking can be started after heating for 20s.

Example 2

[0031] Fiber activated carbon having a fiber length of 1mm was added to 100°C hot water and boiled for 60min, followed by filtration, washing and drying at a high temperature of 150°C with ventilation.

[0032] By mass percentage, tobacco pulp 50%, wood pulp 17%, fiber-activated carbon slurry 25% and Arabic gum 8% were mixed well and fed directly into the folder net paper machine for shaping. The tobacco substrate was obtained by drying with a hot-air dryer, and the resulting tobacco substrate had a thickness of 150 μ m and thickness weight (g) of 80 g/m².

[0033] Coating liquid comprising, by mass percentage, flue-cured tobacco essential oil 10%, tobacco flavor 20% and glycerol 70% was prepared. 35% of the coating liquid based on the mass percentage of the tobacco substrate was sprayed onto the tobacco substrate, then balanced in a constant temperature and humidity incubator at an ambient temperature (22 $\pm$ 1) °C and relative humidity (60 $\pm$ 2)% for 48h to obtain tobacco substrate sample 2#.

[0034] The above substrate was rolled and made into a tobacco roll, compounded with a hollow filter rod to form a cigarette and inserted into a cigarette utensil for use. The heating temperature for the cigarette utensil was set to 350°C, and smoking can be started after heating for 10s.

Example 3

[0035] Activated carbon fiber having a fiber length of 5mm was added to 100°C hot water and boiled for 60min, followed by filtration, washing and drying at a high temperature of 150°C with ventilation.

[0036] By mass percentage, tobacco pulp 76.5%, wood pulp 9.5%, activated carbon fiber slurry 10% and guar gum 4% were mixed well and fed directly into the folder net paper machine for shaping. The tobacco substrate was obtained by drying with a hot-air dryer, and the resulting tobacco substrate had a thickness of 150 μ m and thickness weight (g) of 80 g/m².

[0037] Coating liquid, comprising, by mass percent-

age, flue-cured tobacco essential oil 5%, tobacco flavor 15%, propylene glycol 20% and glycerol 60% was prepared. 40% of the coating liquid based on the mass percentage of the tobacco substrate was sprayed onto the tobacco substrate, then balanced in a constant temperature and humidity incubator at an ambient temperature (22 $\pm$ 1) °C and relative humidity (60 $\pm$ 2)% for 48h to obtain tobacco substrate sample 3#.

[0038] The substrate was rolled and made into a tobacco roll, compounded with a hollow filter rod to form a cigarette and inserted into a cigarette utensil for use. The heating temperature for the cigarette utensil was set to 200°C, and smoking can be started after heating for 30s.

15 Comparative Example 1

[0039] The coating liquid was sprayed onto a conventional substrate, and the coating liquid composition and mass percentage were the same as in Example 1. The resulting substrate was rolled and made into a tobacco roll, compounded with a hollow filter rod to form a cigarette and inserted into a cigarette utensil for use. The heating temperature was set to 250°C, and smoking can be started after heating for 20s.

[0040] By sucking the above substrate sample, it can be found that the tobacco substrate to which activated carbon is added can better adsorb the coating liquid, while for the ordinary substrate, it was found that the coating liquid penetrates to the cigarette paper. The tobacco substrate added with activated carbon can releases a larger amount of smog and more abundant tobacco aroma during the suction process. Therefore, adding activated carbon in the substrate can enhance the quality of heat-not-burn tobacco substrate, having a better application prospect.

[0041] The above embodiments are merely illustrative of the present disclosure and are not intended to limit the present disclosure. While the present disclosure has been described in detail with reference to the embodiments, it will be understood by those skilled in the art that various combinations, modifications, or equivalents of the technical solutions of the present disclosure are not to be regarded as a departure from the spirit and scope of the technical solutions of the present disclosure, and are regarded within the scope of the claims of the present disclosure.

Claims

1. A method for preparing heat-not-burn tobacco substrate containing activated carbon, comprising the following steps:

Step 1 - Preparation of activated carbon slurry: activated carbon is weighed, mixed with water, deflaked and pulped, to make an activated carbon slurry with a concentration of 1~5%;

Step 2 - Preparation of tobacco slurry: tobacco raw material is weighed and put into water for soaking; filtration is carried out after soaking, and the filtered tobacco is mechanically pulped to obtain a tobacco slurry;

Step 3 - Preparation of tobacco substrates: by weight parts, 50~80 parts of tobacco slurry, 8~25 parts of wood pulp, 10~30 parts of activated carbon slurry and 4~8 parts of binder are weighted and mixed well; the mixed material is fed into a paper machine to form a wet tobacco substrate and dried by hot air to obtain a tobacco substrate;

Step 4 - Preparation of coating liquid: by weight parts, 1~40 parts of tobacco essential oil, 2~20 parts of tobacco flavor and 50~80 parts of atomizing agent are weighted and mixed well to obtain a coating liquid;

Step 5 - Coating tobacco substrates: the coating liquid obtained in Step 4 is weighed according to the weight of 25~40% of the tobacco substrate obtained in Step 3 and sprayed onto the tobacco substrate; the tobacco substrate is allowed to stand for 40 to 48 hours under constant temperature and humidity conditions to obtain a heat-not-burn tobacco substrate containing activated carbon.

2. The method for preparing heat-not-burn tobacco substrate containing activated carbon according to claim 1, **characterized in that**, in Step 1, the activated carbon are activated carbon particles and/or activated carbon fibers; the activated carbon particles have a particle size of 80~100 meshes; and the activated carbon fibers have a fiber length of 0.5~8mm.
3. The method for preparing heat-not-burn tobacco substrate containing activated carbon according to claim 1, **characterized in that**, in Step 2, the tobacco material comprises one or more of tobacco stem, tobacco powder, crumbled tobacco leaf and tobacco shred.
4. The method for preparing heat-not-burn tobacco substrate containing activated carbon according to claim 1, **characterized in that**, in Step 3, the wood pulp is one or more of spruce bleached kraft pulp, poplar bleached kraft pulp, needle leave wood bleached kraft pulp and eucalyptus bleached kraft pulp; the binder is one or more of xanthate gum, arabic gum and guar gum.
5. The method for preparing heat-not-burn tobacco substrate containing activated carbon according to claim 1, **characterized in that**, in Step 3, the obtained tobacco substrates have a moisture content of 10~15%, a thickness of 10~300 μ m and a thick-

ness weight (g) of 20~200 g/m².

6. The method for preparing heat-not-burn tobacco substrate containing activated carbon according to claim 1, **characterized in that**, in Step 4, the tobacco essential oil is prepared by the following method: tobacco leaf or tobacco shred is pulverized by a pulverizer and passed through a 40~100 mesh sieve; a certain ratio of solvent water, ethanol or ethyl ether is added for ultrasonic extraction at a certain temperature for 2~10h; after sedimentation, filtration and concentration under reduced pressure, tobacco primary extract is obtained and subjected to molecular distillation for fine extraction to obtain tobacco essential oil; the conditions for the molecular distillation are as following: feed rate 2~10mL/min, vacuum degree 15~20Pa, injection temperature 80°C, heating temperature 60~80°C, cooling temperature 10~20°C, and rotation speed 300~400r/min.
7. The method for preparing heat-not-burn tobacco substrate containing activated carbon according to claim 1, **characterized in that**, in Step 4, the tobacco flavor is one or more of vanillin, benzoic acid, phenylacetic acid, 10% Peru extractum, 5% tree moss extractum, 90% clove oil, anisaldehyde, 10% pineapple ketone, benzyl alcohol, phenylethyl alcohol, acetoltoluene, ethyl phenylacetate, plum extractum, phenylacetaldehyde, acetophenone, angelica lactone, farnesyl ketone, dodecanoic acid, isovaleric acid, benzyl butyrate, octanol, tetramethylpyrazine, citric acid, ethyl maltol, sclareol, sclareolide, ambergris ether, davana oil, orange leaf oil, valerian oil, vetiver oil, labdanum extractum, storax extractum, angelica root tincture, extractum tamarind liquidum, tamarind extractum, chamomile extractum, vanilla extractum, fig extractum, apple juice, megastigmatrienone, dihydrokiwifruittactone, β -ionone, palmitic acid and geranyl acetone.
8. The method for preparing heat-not-burn tobacco substrate containing activated carbon according to any one of claims 1 to 7, **characterized in that**, in Step 4, the atomizing agent is one or more of propylene glycol, triethylene glycol, 1,3-butanediol, glycerol, dipropylene glycol and polyethylene glycol.
9. A tobacco substrate prepared by the method for preparing heat-not-burn tobacco substrate containing activated carbon according to any one of claims 1 to 8.
10. Use of the tobacco substrate prepared by the method for preparing heat-not-burn tobacco substrate containing activated carbon according to claim 9, **characterized in that**, the tobacco substrate prepared by the method according to any one of claims 1 to 8 is rolled and made into a cigarette, placed in a cig-

arete utensil which is set with a heating temperature 200~500°C, a heating time 10~30s, and smoking can be started after the heating time.

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2016/108256

A. CLASSIFICATION OF SUBJECT MATTER

B24B 15/00 (2006.01) i

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

B24B

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

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

CNABS, CNTXT, VEN: charcoal, tobacco+, cigar+, no? w burn+, no? w combust, active? w carbon, coat+

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
PX	CN 105747264 A (CHINA TOBACCO GUIZHOU INDUSTRIAL CO., LTD.), 13 July 2016 (13.07.2016), claims 1-10	1-10
A	CN 103892442 A (CHINA TOBACCO GUANGDONG INDUSTRIAL CO., LTD.), 02 July 2014 (02.07.2014), claim 1, and description, paragraphs 14-28	1-10
A	CN 103929984 A (PHILIP MORRIS PRODUCTS S.A.), 16 July 2014 (16.07.2014), the whole document	1-10
A	CN 101268859 A (ZHENGZHOU TOBACCO RESEARCH INSTITUTE OF CNTC), 24 September 2008 (24.09.2008), the whole document	1-10
A	JP 5857210 B2 (TOYOCO KK), 19 December 1983 (19.12.1983), the whole document	1-10

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

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Date of the actual completion of the international search

13 February 2017 (13.02.2017)

Date of mailing of the international search report

02 March 2017 (02.03. 2017)

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

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/CN2016/108256

Patent Documents referred in the Report	Publication Date	Patent Family	Publication Date
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REFERENCES CITED IN THE DESCRIPTION

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- CN 103181613 [0004]