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(54) **METHOD FOR MANUFACTURING FRAGRANCE-CARRYING TOBACCO FILLER, FRAGRANCE-CARRYING TOBACCO FILLER, AND HEATING TYPE FLAVOR INHALER**

(57) A method of producing a flavorant-carrying tobacco filler comprises extracting a water-soluble component contained in a tobacco material with an aqueous solvent from the tobacco material to obtain a tobacco extraction liquid and a tobacco residue, heating the tobacco residue to form a carboxyl group in the tobacco residue, and mixing the tobacco residue having the carboxyl group formed therein with a flavorant having a hydroxyl group to cause an esterification reaction between the carboxyl group and the hydroxyl group, thereby obtaining a flavorant-carrying tobacco residue.

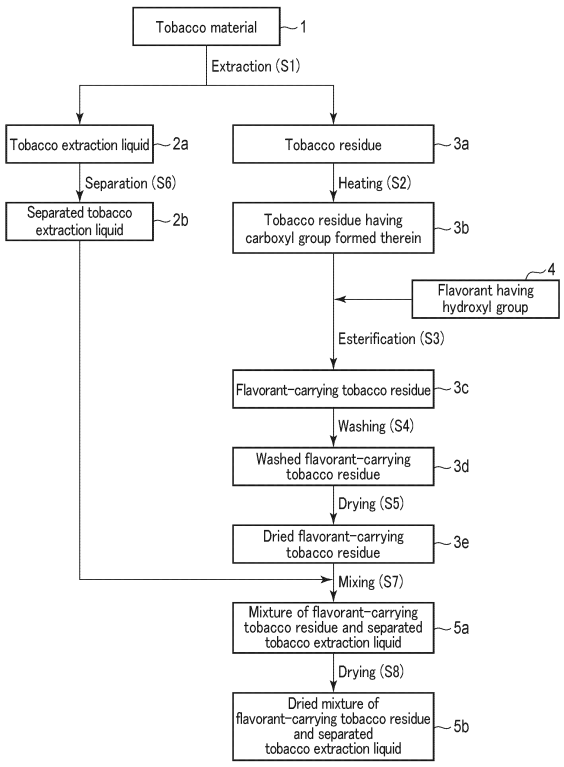


FIG. 1

EP 3 711 494 A1

Description

FIELD

[0001] The present invention relates to a method of producing a flavorant-carrying tobacco filler, a flavorant-carrying tobacco filler, and a heating type flavor inhaler.

BACKGROUND

[0002] For tobacco products, there are known a combustion type smoking article (e.g., cigarette) which provides a user with tobacco flavor by burning a tobacco filler such as cut tobacco, and a heating type flavor inhaler which provides a user with tobacco flavor by heating without burning a tobacco filler.

[0003] There have been improvements in tobacco fillers used for tobacco products to facilitate a release of tobacco flavor so that the user can taste adequate tobacco flavor. For example, WO 2013/098920 discloses that a content of ester flavor components and components contributing to tobacco flavor and taste contained in cut tobacco are increased by aging the cut tobacco, which is to be used in manufacture of a tobacco product immediately thereafter, under a sealed condition in an atmospheric environment, and then adding a polyol to the cut tobacco.

SUMMARY

TECHNICAL PROBLEM

[0004] The heating type flavor inhaler does not burn the tobacco filler, and therefore sufficient tobacco flavor is less likely to be provided to the user as compared to the combustion type smoking article. Thus, an object of the present invention is to provide a flavorant-carrying tobacco filler which has, when incorporated into a heating type flavor inhaler, excellent flavorant storage stability and excellent flavorant release properties.

[0005] According to the first aspect, there is provided a method of producing a flavorant-carrying tobacco filler, the method comprising:

extracting a water-soluble component contained in a tobacco material with an aqueous solvent from the tobacco material to obtain a tobacco extraction liquid and a tobacco residue;
heating the tobacco residue to form a carboxyl group in the tobacco residue; and
mixing the tobacco residue having the carboxyl group formed therein with a flavorant having a hydroxyl group to cause an esterification reaction between the carboxyl group and the hydroxyl group, thereby obtaining a flavorant-carrying tobacco residue.

[0006] According to the second aspect, there is provided a flavorant-carrying tobacco filler obtainable by the above-mentioned method.

[0007] According to the third aspect, there is provided a heating type flavor inhaler, comprising a tobacco flavor source, the tobacco flavor source including:

the above-mentioned flavorant-carrying tobacco filler; and
an aerosol source mixed with the flavorant-carrying tobacco filler.

ADVANTAGEOUS EFFECTS OF INVENTION

[0008] According to the present invention, it is possible to provide a flavorant-carrying tobacco filler which has, when incorporated into a heating type flavor inhaler, excellent flavorant storage stability and excellent flavorant release properties.

BRIEF DESCRIPTION OF THE DRAWINGS

[0009]

FIG. 1 is a flowchart showing a preferred embodiment of a method according to the present invention;
FIG. 2 illustrates a flavorant carrying reaction and a flavorant release reaction;
FIG. 3 is a partially cutaway view illustrating an example of a heating type flavor inhaler;
FIG. 4 illustrates a flavorant existence state in a tobacco pod;

FIG. 5 is a graph showing an amount of flavorant carried of a flavorant-carrying tobacco filler;
FIG. 6 illustrates results of analysis by Fourier transform infrared spectroscopy; and
FIG. 7 is a graph showing an influence of heating conditions of heating step (S2) on the amount of flavorant carried.

DETAILED DESCRIPTION

[0010] Hereinafter, the present invention will be described, but the following description is for the purpose of detailed explanation of the present invention, and is not intended to limit the present invention.

<1. Method of Producing Flavorant-carrying Tobacco Filler>

[0011] According to an embodiment, a method of producing a flavorant-carrying tobacco filler includes:

(S1) extracting a water-soluble component contained in a tobacco material with an aqueous solvent from the tobacco material to obtain a tobacco extraction liquid and a tobacco residue;
(S2) heating the tobacco residue to form a carboxyl group in the tobacco residue; and
(S3) mixing the tobacco residue having the carboxyl group formed therein with a flavorant having a hydroxyl group to cause an esterification reaction between the carboxyl group and the hydroxyl group, thereby obtaining a flavorant-carrying tobacco residue.

[0012] The above-described embodiment may further include:

(S6) separating and removing the solvent from the tobacco extraction liquid to prepare a separated tobacco extraction liquid; and
(S7) mixing the separated tobacco extraction liquid with the flavorant-carrying tobacco residue to obtain a mixture of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid.

[0013] According to a preferred embodiment, a method of producing a flavorant-carrying tobacco filler includes:

(S1) extracting a water-soluble component contained in a tobacco material with an aqueous solvent from the tobacco material to obtain a tobacco extraction liquid and a tobacco residue;
(S2) heating the tobacco residue to form a carboxyl group in the tobacco residue;
(S3) mixing the tobacco residue having the carboxyl group formed therein with a flavorant having a hydroxyl group to cause an esterification reaction between the carboxyl group and the hydroxyl group, thereby obtaining a flavorant-carrying tobacco residue;
(S4) washing the flavorant-carrying tobacco residue to wash away an unesterified flavorant; and
(S5) drying the washed flavorant-carrying tobacco residue to obtain a dried flavorant-carrying tobacco residue.

[0014] The above-described preferred embodiment may further include:

(S6) separating and removing the solvent from the tobacco extraction liquid to prepare a separated tobacco extraction liquid;
(S7) mixing the separated tobacco extraction liquid with the dried flavorant-carrying tobacco residue to obtain a mixture of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid; and
(S8) drying the mixture to obtain a dried mixture.

[0015] The above-described preferred embodiment is shown in FIG. 1. Hereinafter, the preferred embodiment is described with reference to FIG. 1 in the order of steps (S1) to (S8).

[Extraction step (S1)]

[0016] A water-soluble component contained in a "tobacco material (1)" is extracted with an aqueous solvent from the tobacco material to obtain a "tobacco extraction liquid (2a)" and a "tobacco residue (3a)" (see FIG. 1).

[0017] As the tobacco material (1), cut tobacco which is ready to be incorporated into a tobacco product such as a smoking article or a flavor inhaler can be used. The "cut tobacco which is ready to be incorporated into a tobacco product" refers to cut tobacco which is ready to be incorporated into a tobacco product through various processes including a drying process of harvested tobacco leaves in a farm house, thereafter an aging process for one to several years in a leaf processing facility, and thereafter blending and cutting processes in a manufacturing facility.

[0018] The cut tobacco may be any of cut pieces of stemmed leaves, cut pieces of midrib, cut pieces of reconstituted tobacco (i.e., a tobacco material obtained by processing leaf scraps, cut tobacco scraps, midrib scraps, and fine powder generated in the plant processes into a reusable shape), or a mixture thereof. The pulverized product obtained by pulverizing the cut tobacco may be used for the extraction in order to increase an extraction efficiency.

[0019] As the cut tobacco, cut tobacco derived from any tobacco variety can be used. For example, cut tobacco derived from flue-cured tobacco, Burley tobacco, Oriental tobacco or the like can be used. As the cut tobacco, cut tobacco derived from a single variety may be used, or a mixture of cut tobacco derived from different varieties may be used.

[0020] As the aqueous solvent, water or water-containing ethanol can be used. As the water-containing ethanol, for example, a mixture of ethanol and water at a volume ratio of 1:1 can be used. The aqueous solvent is generally water, preferably water of room temperature (e.g., approximately 20°C) to 70°C. The aqueous solvent can be used, for example, in an amount of 500 to 5000% by mass with respect to the tobacco material (1).

[0021] Extraction can be performed by, for example, immersing the tobacco material (1) in warm water at 40 to 60°C for 30 to 180 minutes, or by shaking the tobacco material (1) (e.g., 200 rpm) in warm water at 40 to 60°C for 30 to 180 minutes.

[0022] Extraction may also be performed by repeating the extracting process multiple times. Specifically, extraction may be performed by extracting the water-soluble component contained in the tobacco material (1) with the aqueous solvent from the tobacco material, and then placing the resulting tobacco residue (3a) in a new aqueous solvent to perform the second extracting process, and as necessary, repeating the extracting process with a new aqueous solvent.

[0023] Before extraction with the aqueous solvent, an additional step may be performed to extract with an organic solvent, from the tobacco material (1), an organic solvent-soluble component contained in the tobacco material. Specifically, this additional step can be performed by immersing the tobacco material in the organic solvent (e.g., hexane, diethyl ether, etc.) for 30 to 180 minutes, or shaking the tobacco material in the organic solvent (e.g., hexane, diethyl ether, etc.) for 30 to 180 minutes. This additional step allows extraction of the organic solvent-soluble component contained in the tobacco material. More specifically, by performing this additional step, it is possible to collect, in the organic solvent, flavor components having a lower polarity and insoluble in water, out of components contained in the tobacco material. The extraction liquid obtained by the additional step can be mixed with a flavorant-carrying tobacco residue in a later-described mixing step (S7).

[0024] The extraction step (S1) yields a mixture of a tobacco extraction liquid (2a) containing the water-soluble component contained in the tobacco material (1), and a tobacco residue (3a). The water-soluble component contained in the tobacco material (1) includes, for example, organic acids, specifically formic acid, acetic acid, propionic acid, etc. Thus, the tobacco residue (3a) substantially contains no water-soluble components contained in the tobacco material.

[0025] The tobacco extraction liquid (2a) and the tobacco residue (3a) are separated, and the tobacco residue (3a) is used as a substrate for carrying a flavorant in subsequent steps.

[0026] The tobacco residue (3a) may be used as a substrate for carrying a flavorant after being dried, or the tobacco residue may be molded into a molding to use the tobacco residue molding as a substrate for carrying the flavorant. The tobacco residue molding may be a sheet-like molding, or cut pieces obtained by cutting the sheet-like molding into a shape of cut tobacco, or powder obtained by pulverizing the sheet-like molding. The sheet-like molding can be prepared by, for example, molding the tobacco residue into a sheet shape using a paper-making technique.

[Heating step (S2)]

[0027] The "tobacco residue (3a)" obtained in the extraction step (S1) is heated to form a carboxyl group in the tobacco residue (3a), thereby obtaining a "tobacco residue having the carboxyl group formed therein (3b)" (see FIG. 1).

[0028] As described above, the tobacco residue (3a) may be obtained by drying the tobacco residue obtained in the extraction step (S1), or may be a tobacco residue molding.

[0029] In the heating step (S2), the heating temperature may be any temperature as long as a carboxyl group can be formed on a surface of the tobacco residue. Heating time can be set as appropriate depending on the heating temperature, and in general, the lower the heating temperature becomes, the longer the heating time needs to be set.

[0030] Heating can be performed by heating the tobacco residue (3a) at a temperature of, for example, 150 to 300°C, preferably 170 to 300°C, more preferably 170 to 270°C, still more preferably 190 to 250°C, and most preferably 230°C.

[0031] Heating can be performed for, for example, 0.5 to 6 hours, preferably 1 to 3 hours. For example, if heating is performed at 150°C, heating for 3 hours is superior in carboxyl group formation to heating for 1 hour.

[0032] A heating temperature lower than 150°C decreases the efficiency of forming the carboxyl group on the surface of the tobacco residue. On the other hand, if the heating temperature is higher than 300°C, carbonization progresses with combustion under the condition where oxygen coexists, and the amount of carboxyl groups formed decreases easily because of progress of reactions of decarboxylation, etc. Furthermore, if the heating temperature is higher than 300°C, the efficiency of forming the carboxyl groups decreases under the condition where oxygen does not coexist.

[0033] Heating can be performed under a sealed condition with the tobacco residue (3a) placed in a container having

a lid.

[0034] By heating, cell wall components such as cellulose contained in the tobacco residue (3a) can be pyrolyzed, thereby forming a carboxyl group. In this manner, the amount of carboxyl groups on cell wall surfaces of cells constituting the tobacco residue (3a) can be increased. Such heating is also called "partial carbonization", and it is known that the partial carbonization of plant cells increases the amounts of carboxyl groups and phenolic hydroxyl groups as functional groups on the cell wall surfaces of the plant cells.

[0035] It is preferable that the tobacco residue having the carboxyl group formed therein (3b) be cooled to a temperature lower than a reaction temperature of the next esterification step (S3) and transferred to the next esterification step (S3). This will prevent decomposition of the flavorant used in the next esterification step (S3).

[Esterification step (S3)]

[0036] The "tobacco residue having the carboxyl group formed therein (3b)" obtained in the heating step (S2) is mixed with a "flavorant (4) having a hydroxyl group" to cause an esterification reaction between the carboxyl group and the hydroxyl group, thereby obtaining a "flavorant-carrying tobacco residue (3c)" (see FIG. 1).

[0037] As the flavorant (4) having a hydroxyl group, any flavorant having a hydroxyl group can be used, specifically an alcoholic flavorant can be used. Examples of the flavorant (4) having a hydroxyl group include menthol, benzyl alcohol, phenethyl alcohol, ethanol, hexanol, hexenol, linalool, coniferyl alcohol, sinapyl alcohol, cinnamyl alcohol, homovanillyl alcohol, and the like. The flavorant (4) having a hydroxyl group may be a single flavorant, or a mixture of multiple flavorants. In addition, a natural material such as an extract or essential oil containing one or more of these flavorant components can be used as the flavorant (4) having a hydroxyl group. Examples of the natural material such as an extract or essential oil include peppermint oil, spearmint oil, a licorice extract, a coffee extract, a cinnamon extract, and the like. In the following description, the flavorant (4) having a hydroxyl group is simply referred to as a flavorant as well.

[0038] The esterification reaction can be performed according to publicly-known reaction conditions. Specifically, the esterification reaction can be performed by reacting the tobacco residue having the carboxyl group formed therein (3b) and the flavorant (4) having a hydroxyl group at a temperature of 80 to 140°C under acidic conditions. Here, the acidic conditions are, for example, pH 2 or below, preferably pH 0.5 to 1.5. The temperature of the esterification reaction is preferably 100 to 120°C. If the temperature of the esterification reaction is too high, another reaction may occur.

[0039] The time of the esterification reaction can be set as appropriate depending on the temperature of the esterification reaction, and in general, the lower the reaction temperature becomes, the longer the reaction time needs to be set.

[0040] The esterification reaction can be performed for, for example, 0.5 to 3 hours, preferably 1 to 2 hours. In the esterification reaction, for example, 0.1 to 10 g of the flavorant can be reacted with 1 g of the tobacco residue having the carboxyl group formed therein (3b).

[0041] Preferably, the esterification reaction can be performed for 1 to 2 hours at a temperature of 100 to 120°C under the conditions of pH 0.5 to 1.5.

[0042] The esterification reaction is a reversible reaction, and thus in order to promote ester formation, it is preferable to perform the esterification reaction under conditions where no water exists or a small amount of water exists. Therefore, it is preferable to add a small amount of a sulfuric acid aqueous solution to the flavorant, make adjustments to the above-described pH, and use the resulting sulfuric acid-containing flavorant liquid as a reactive solvent of the esterification reaction.

[0043] The esterification reaction generates an ester between the carboxyl group of the tobacco residue and the hydroxyl group of the flavorant, and as a result, the flavorant is carried on the tobacco residue.

[0044] As described above, the tobacco residue substantially contains no water-soluble component (e.g., organic acid) contained in the tobacco material. Therefore, in the esterification reaction, the hydroxyl group of the flavorant does not react with the carboxyl group contained in the water-soluble component of the tobacco material, and can efficiently react with the carboxyl group present on the surface of the tobacco residue. Thus, in the method according to the present invention, the water-soluble component contained in the tobacco material is removed in the extraction step (S1), and the amount of carboxyl groups is increased on the surface of the tobacco residue in the heating step (S2), and therefore, the flavorant-carrying efficiency can be enhanced in the esterification step (S3).

[Washing step (S4)]

[0045] After the esterification step (S3), the "flavorant-carrying tobacco residue (3c)" is washed to wash away an unesterified flavorant, thereby obtaining a "washed flavorant-carrying tobacco residue (3d)" (see FIG. 1). The washing step (S4) may or may not be performed.

[0046] The washing step (S4) can be performed by, after the esterification reaction, dividing the reaction mixture into a liquid part and a solid part (i.e., the flavorant-carrying tobacco residue (3c)), and pouring over the flavorant-carrying tobacco residue (3c) an organic solvent capable of dissolving the flavorant, such as ethanol. Alternatively, the washing

step can be performed by adding the organic solvent to the flavorant-carrying tobacco residue (3c), and immersing or shaking the flavorant-carrying tobacco residue (3c) in the organic solvent.

[0047] As a flavorant, if a flavorant which is solid at room temperature, such as menthol, is used, when the reaction mixture cools after the esterification reaction, the flavorant contained in the reaction mixture may be solidified. Thus, in this case, it is preferable that after the esterification reaction, the reaction mixture be divided into a liquid part and a solid part (i.e., the flavorant-carrying tobacco residue (3c)) before being cooled to, for example, 60°C or less.

[0048] On the other hand, an organic solvent capable of dissolving the flavorant, such as ethanol, may induce a transesterification reaction in the flavorant carried on the tobacco residue, resulting in a removal of the flavorant from the tobacco residue. For this reason, when an organic solvent capable of dissolving the flavorant is applied to the flavorant-carrying tobacco residue (3c), it is preferable that the flavorant-carrying tobacco residue (3c) be cooled to, for example, 60°C or less, preferably to room temperature (e.g., 15 to 25°C).

[0049] By the washing step (S4), it is considered that the flavorant carried via an ester bond on the tobacco residue is not washed away while the flavorant adhering to the tobacco residue is partially washed away with the rest remaining adhered. In the present specification, "adhering" refers to the state in which the flavorant is physically carried on the surface of the tobacco residue. The surface of the tobacco residue includes, in addition to a surface of a flat plane, a surface having pores produced when the partial carbonization occurs in the heating step (S2) .

[0050] Whether the flavorant adhering to the tobacco residue is washed away or remains adhered varies depending on the degree of washing (specifically, the method of washing, the number of washing times, the washing time, the amount of organic solvent used for washing, etc.). That is, as the degree of washing becomes lower, the ratio of the flavorant washed away decreases, while the ratio of the flavorant remaining adhered to the tobacco residue increases. Conversely, as the degree of washing becomes higher, the ratio of the flavorant washed away increases, while the ratio of the flavorant remaining adhered to the tobacco residue decreases.

[Drying step (S5)]

[0051] The "washed flavorant-carrying tobacco residue (3d)" obtained in the washing step (S4) is dried to obtain a "dried flavorant-carrying tobacco residue (3e)" (see FIG. 1). If the washing step (S4) is not performed, the "flavorant-carrying tobacco residue (3c)" obtained in the esterification step (S3) is dried to obtain a "dried flavorant-carrying tobacco residue (3e)". The drying step (S5) may or may not be performed.

[0052] The drying step (S5) may be performed by using a dryer or by natural drying. For example, natural drying may be performed by allowing the flavorant-carrying tobacco residue (3c or 3d) to stand for 1 to 7 days under conditions of a temperature of 5 to 40°C and a humidity of 10 to 90%. When a dryer is used, drying may be performed by vacuum drying for 1 to 5 hours while avoiding heating and keeping the temperature at 40°C or below.

[0053] The "dried flavorant-carrying tobacco residue (3e)" can be incorporated into a heating type flavor inhaler as a tobacco filler. Alternatively, the "dried flavorant-carrying tobacco residue (3e)" may be mixed with a separated tobacco extraction liquid (2b) in a later-described mixing step (S7).

[0054] If the drying step (S5) is not performed, the "washed flavorant-carrying tobacco residue (3d)" may be incorporated into a heating type flavor inhaler as a tobacco filler, or may be mixed with the separated tobacco extraction liquid (2b) in the later-described mixing step (S7) .

[0055] When neither the washing step (S4) nor the drying step (S5) is performed, the "flavorant-carrying tobacco residue (3c)" may be incorporated into a heating type flavor inhaler as a tobacco filler, or may be mixed with the separated tobacco extraction liquid (2b) in the later-described mixing step (S7).

[Separation step (S6)]

[0056] A solvent is separated and removed from the "tobacco extraction liquid (2a)" obtained in the extraction step (S1) to prepare a "separated tobacco extraction liquid (2b)" (see FIG. 1).

[0057] The separation step (S6) may be performed by, for example, evaporating and removing the solvent from the tobacco extraction liquid, specifically by vacuum distillation using an evaporator.

[0058] Separation can be performed so as to obtain 0.5 to 5 mL of the separated tobacco extraction liquid (2b) per 1 g of the tobacco material (1). For example, if the aqueous solvent is used in an amount of 1000% by mass with respect to the tobacco material (1) in the extraction step (S1), separation may be performed until the amount of the tobacco extraction liquid is about one tenth to one fifth.

[Mixing step (S7)]

[0059] The "separated tobacco extraction liquid (2b)" obtained in the separation step (S6) is mixed with the "dried flavorant-carrying tobacco residue (3e)" obtained in the drying step (S5) to obtain a "mixture (5a) of the flavorant-carrying

tobacco residue and the separated tobacco extraction liquid" (see FIG. 1).

[0060] As described above, if the drying step (S5) is not performed, the "separated tobacco extraction liquid (2b)" is mixed with the "washed flavorant-carrying tobacco residue (3d)" to obtain a "mixture (5a) of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid". If neither the washing step (S4) nor the drying step (S5) is performed, the "separated tobacco extraction liquid (2b)" is mixed with the "flavorant-carrying tobacco residue (3c)" to obtain a "mixture (5a) of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid".

[0061] It is preferable that the "separated tobacco extraction liquid (2b)" and the "flavorant-carrying tobacco residue (any of 3c, 3d and 3e)" used in the mixing step (S7) be derived from the "tobacco extraction liquid (2a)" and the "tobacco residue (3a)", respectively, obtained in the same extraction step (S1). In this case, mixing the "separated tobacco extraction liquid (2b)" and the "flavorant-carrying tobacco residue (any of 3c, 3d and 3e)" reproduces constituents of the "tobacco material (1)" as a starting material.

[0062] In the mixing step (S7), in either case, it should be noted that the content or content ratio of the smoking flavor components contained in the "mixture (5a) of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid" obtained by mixing the "flavorant-carrying tobacco residue (any of 3c, 3d, and 3e)" and the "separated tobacco extraction liquid (2b)" is equal to or less than the content or content ratio of the smoking flavor components contained in the "tobacco material (1)" used to prepare the "flavorant-carrying tobacco residue". To accomplish this, for example, the amount of the "separated tobacco extraction liquid (2b)" used in the mixing step (S7) may be set not to exceed the total amount of the "separated tobacco extraction liquid (2b)" obtained from the "tobacco material (1)" used to prepare the "flavorant-carrying tobacco residue" used in the mixing step (S7).

[Drying step (S8)]

[0063] The "mixture (5a) of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid" obtained in the mixing step (S7) is dried to obtain a "dried mixture (5b) of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid" (see FIG. 1). The drying step (S8) may or may not be performed.

[0064] When the drying step (S8) is performed, it is preferable to complete the drying step in a short period of time. Drying can be performed by vacuum drying using an evaporator or a centrifugal evaporator, or by freeze drying. Alternatively, drying can be performed by heat drying at a temperature of 50 to 70°C for 0.5 to 2 hours.

[0065] The "dried mixture (5b) of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid" can be incorporated into a heating type flavor inhaler as a tobacco filler.

[0066] If the drying step (S8) is not performed, the "mixture (5a) of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid" can be incorporated into a heating type flavor inhaler as a tobacco filler.

<2. Flavorant-carrying Tobacco Filler>

[0067] According to another aspect, there is provided a flavorant-carrying tobacco filler obtainable by the above-described "method of producing a flavorant-carrying tobacco filler".

[0068] Specifically, the flavorant-carrying tobacco filler is any one of the above-described "flavorant-carrying tobacco residue (3c)", "washed flavorant-carrying tobacco residue (3d)", "dried flavorant-carrying tobacco residue (3e)", "mixture (5a) of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid", and "dried mixture (5b) of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid" (see FIG. 1). Thus, the "flavorant-carrying tobacco filler" in the present specification encompasses the above-described five types of products as concrete examples.

[0069] Preferably, the flavorant-carrying tobacco filler is either the "dried flavorant-carrying tobacco residue (3e)" or the "dried mixture (5b) of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid", produced by the above-described method. More preferably, the flavorant-carrying tobacco filler is the "dried mixture (5b) of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid" produced by the above-described method.

[0070] FIG. 2 schematically shows the flavorant carrying reaction occurring in the heating step (S2) and the esterification step (S3) of the above-described method.

[0071] FIG. 2 shows that in the heating step (S2), when the "tobacco residue (3a)" is heated, a carboxyl group (-COOH) is formed in the tobacco residue, thereby obtaining the "tobacco residue having the carboxyl group formed therein (3b)". FIG. 2 further shows that in the subsequent esterification step (S3), when the "tobacco residue having the carboxyl group formed therein (3b)" and the "flavorant (R'OH) (4) having a hydroxyl group" are reacted, an esterification reaction occurs between the carboxyl group and the hydroxyl group, thereby obtaining the "flavorant-carrying tobacco residue (3c)".

[0072] Therefore, the flavorant-carrying tobacco filler includes the following when specified by its structure:

a tobacco residue; and
a flavorant carried on the tobacco residue by an ester bond between the carboxyl group of the tobacco residue and

the hydroxyl group of the flavorant.

[0073] Preferably, the flavorant-carrying tobacco filler includes:

a tobacco residue;
a flavorant carried on the tobacco residue by an ester bond between the carboxyl group of the tobacco residue and the hydroxyl group of the flavorant; and
a component contained in a tobacco extraction liquid.

[0074] Here, it is preferable that the "tobacco extraction liquid" be a "tobacco extraction liquid" obtained at the time of water extraction of the tobacco material performed to prepare the "tobacco residue". Therefore, the "component contained in a tobacco extraction liquid" can be rephrased as a water-soluble component of the tobacco material. Specifically, the "component contained in a tobacco extraction liquid" may be a "separated tobacco extraction liquid" obtained by extracting the tobacco material with the aqueous solvent and separating and removing the solvent from the tobacco extraction liquid, or may be a "dried separated tobacco extraction liquid" obtained by drying the separated tobacco extraction liquid. Here, the "tobacco material" is as defined in the present specification.

[0075] However, since the flavorant-carrying tobacco filler has acid functional groups (e.g., a hydroxyl group) formed therein other than the carboxyl group in the heating step (S2), it is difficult to completely specify the structure thereof.

[0076] In the flavorant-carrying tobacco filler, it is preferable that the flavorant be carried on the tobacco residue in an amount of 0.01 mg or more with respect to 1 g of the tobacco residue. In the flavorant-carrying tobacco filler, it is more preferable that the flavorant be carried on the tobacco residue in an amount of 0.01 to 50 mg with respect to 1 g of the tobacco residue. The amount of flavorant carried represents the total amounts of the flavorant chemically bonded to the tobacco residue by an ester bond and the flavorant physically bonded to the tobacco residue by adhesion.

<3. Heating Type Flavor Inhaler>

[0077] According to still another aspect, there is provided a heating type flavor inhaler provided with a tobacco flavor source that includes the above-described flavorant-carrying tobacco filler and an aerosol source mixed with the flavorant-carrying tobacco filler.

[0078] In the present specification, the aerosol source is a liquid for generating aerosol by heating, and has a hydroxyl group. The aerosol source having a hydroxyl group (hereafter simply referred to as "aerosol source") is, for example, propylene glycol, glycerin, or a mixture thereof.

[0079] Because the flavorant-carrying tobacco filler is incorporated into the heating type flavor inhaler with the aerosol source being mixed, when the heating type flavor inhaler is heated, a transesterification reaction occurs between the carried flavorant and the aerosol source, and the flavorant is released.

[0080] FIG. 2 schematically shows the flavorant release reaction.

[0081] FIG. 2 shows that when the transesterification reaction occurs between the "flavorant-carrying tobacco filler (3c, 3d, 3e, 5a, 5b)" and the "aerosol source (propylene glycol and/or glycerin (PG/G)", the "flavorant (R'OH) (4) having a hydroxyl group" is released, and a "tobacco residue on which the aerosol source is carried" is formed.

[0082] Thus, the aerosol source functions as a supply source for generating aerosol that is inhaled by a user, and also functions as a substrate of the transesterification reaction, thereby involving in the flavorant release. Therefore, the aerosol source needs to be present in the heating type flavor inhaler together with the flavorant-carrying tobacco filler.

[0083] Since the transesterification is a reversible reaction, in order to promote the transesterification reaction in a direction of releasing the flavorant, it is preferable that the aerosol source be present in a predetermined amount or more with respect to the flavorant-carrying tobacco filler. Specifically, it is preferable that the aerosol source be contained in an amount of 50 mg or more per 1 g of the flavorant-carrying tobacco filler in the heating type flavor inhaler. It is more preferable that the aerosol source be contained in an amount of 50 to 2000 mg per 1 g of the flavorant-carrying tobacco filler in the heating type flavor inhaler.

[0084] In the transesterification reaction, the "flavorant (R'OH) having a hydroxyl group" is released, and the carboxyl group (-COOH) of the tobacco residue reacts with the aerosol source (propylene glycol and/or glycerin (PG/G)) to form a -COOPG/G group on the surface of the tobacco residue (see FIG. 2). When such a transesterification reaction occurs, the flavorant carried via the ester bond on the tobacco residue is released by hydrolysis of the ester without being affected by oxidative decomposition or the like.

[0085] The flavorant-carrying tobacco filler may be used as a tobacco filler of a heating type flavor inhaler by combining it with normal cut tobacco on which a flavorant is not carried, or may be used to account for the entire tobacco filler (100% by mass) contained in a heating type flavor inhaler without combining it with normal cut tobacco. In the former case, the flavorant-carrying tobacco filler can be used to account for 1 to 99% by mass of the entire tobacco filler contained in the heating type flavor inhaler. Here, the "normal cut tobacco on which a flavorant is not carried" is the above-described

"cut tobacco which is ready to be incorporated into a tobacco product".

[0086] Hereinafter, a description will be given of a "heating type flavor inhaler" into which the flavorant-carrying tobacco filler according to the present invention can be incorporated.

[0087] The heating type flavor inhaler is an inhaler that provides a user with tobacco flavor by heating, but not burning, the tobacco filler, and various forms are known in the art. The heating type flavor inhaler does not burn a tobacco filler, and is thus referred to as a non-combustion type flavor inhaler as well.

[0088] In the present invention, the heating type flavor inhaler may have any structure as long as the flavorant-carrying tobacco filler can be incorporated therein with an aerosol source being mixed.

[0089] Examples of the heating type flavor inhaler include:

a carbonaceous heat source type inhalation article that heats a mixture of the flavorant-carrying tobacco filler and an aerosol source (hereinafter, referred to as a tobacco flavor source) with combustion heat of a carbon heat source to generate aerosol (see, for example, WO 2006/073065);
an electrical heating type inhalation article provided with an inhaler body containing a tobacco flavor source, and a heating device for electrically heating the inhaler body, where the inhalation article heats the tobacco flavor source with electrical heat to generate aerosol (see, for example, WO 2010/110226); and
an electrical heating type inhalation article provided with a refill type tobacco pod containing a tobacco flavor source, and an inhaler body that heats the tobacco pod with electrical heat to generate aerosol (see, for example, WO 2013/025921).

[0090] In the following, with reference to FIG. 3, a description will be given of an example of a heating type flavor inhaler into which the flavorant-carrying tobacco filler according to the present invention can be incorporated.

[0091] A heating type flavor inhaler 10 shown in FIG. 3 is an electrical heating type inhaler that heats a tobacco flavor source with electrical heat to generate aerosol. In the following description, the heating type flavor inhaler 10 is simply referred to as a flavor inhaler 10.

[0092] The flavor inhaler 10 includes a main body 110 and a mouthpiece 120. The flavor inhaler 10 has a shape extending along a direction in which the main body 110 and the mouthpiece 120 are connected, and includes a non-mouthpiece end (end of the main body 110 side) and a mouthpiece end (end of the mouthpiece 120 side).

[0093] In the following description, if a "non-mouthpiece end side" is referred to for a certain part, the position specified by this "non-mouthpiece end side" indicates the end position of the part closer to the non-mouthpiece end of the flavor inhaler 10. Furthermore, in the following description, if a "mouthpiece end side" is referred to for a certain part, the position specified by this "mouthpiece end side" indicates the end position of the part closer to the mouthpiece end of the flavor inhaler 10.

[0094] The main body 110 includes a tubular body 111, a battery 112, a control circuit 113, and a heater 114.

[0095] The tubular body 111 is a tubular body with a bottom, and is open on the mouthpiece end side so that a later-described tobacco pod 130 can be replaced. The tubular body 111 may be a circular section tubular body or a polygonal section tubular body. The non-mouthpiece end of the tubular body 111 is provided with a charging part (not shown) for charging the battery 112. In addition, a side wall of the tubular body 111 is provided with a power supply button (not shown) to turn on or off the flavor inhaler 10.

[0096] The battery 112 is placed inside the tubular body 111. The battery 112 is, for example, a lithium ion secondary battery. The battery 112 supplies the power necessary to operate the flavor inhaler 10 to the electrical and electronic parts included in the flavor inhaler 10. For example, the battery 112 supplies the power to the control circuit 113 and the heater 114.

[0097] The control circuit 113 is placed in the tubular body 111 between its opening and the battery 112. The control circuit 113 may be placed at another position in the tubular body 111. The control circuit 113 controls the operation of the flavor inhaler 10. Specifically, the control circuit 113 controls the power supplied to the heater 114 based on a value output by a temperature sensor placed in the vicinity of the heater 114.

[0098] The heater 114 is placed on the mouthpiece end side in the tubular body 111. Specifically, the heater 114 is placed in the tubular body 111 between its opening and the control circuit 113. The heater 114 has a cup shape allowing the tobacco pod 130 to be accommodated. The heater 114 is electrically connected to the battery 112 and the control circuit 113. The temperature of the heater 114 is controlled by the control circuit 113. It is preferable that the heater 114 be surrounded by an insulator to avoid its heat transmission to the tubular body 111, the battery 112, the control circuit 113, etc.

[0099] The main body 110 may further include a light-emitting device, for example, on a side wall of the tubular body 111, to notify a user of a heating state of the tobacco pod 130 or a remaining amount of the battery 112.

[0100] Here, the tobacco pod 130 will be described.

[0101] The tobacco pod 130 is placed in the main body 110 to be surrounded by the heater 114. The tobacco pod 130 is replaced by the user after inhalation is performed a predetermined number of times.

[0102] The tobacco pod 130 includes a container 131 and a tobacco flavor source 132.

[0103] The container 131 is made of, for example, a metal (e.g., aluminum).

[0104] The tobacco flavor source 132 is contained in the container 131. The tobacco flavor source 132 includes the above-described flavorant-carrying tobacco filler and aerosol source.

[0105] Before being attached to the main body 110, the tobacco pod 130 is sealed with an aluminum foil lid. When attached to the main body 110, the tobacco pod 130 is unsealed in such a manner that the tobacco flavor can be inhaled from the tobacco flavor source.

[0106] The mouthpiece 120 is provided on the mouthpiece end side of the main body 110 in a detachable manner. The mouthpiece 120 is removed from the main body 110 by the user when the tobacco pod 130 is replaced.

[0107] The mouthpiece 120 includes a protrusion on its non-mouthpiece end side. When the mouthpiece 120 is attached to the main body 110, this protrusion penetrates the lid of the tobacco pod 130 to open the tobacco pod 130. The mouthpiece 120 may not include a protrusion. In this case, the tobacco pod 130 is opened by the user's hand, for example, right before being attached to the main body 110.

[0108] The mouthpiece 120 includes a first gas flow path that leads external air of the flavor inhaler 10 into a space in the tobacco pod 130. The first gas flow path has a gas flow inlet, for example, in the vicinity of the connection between the mouthpiece 120 and the main body 110. The mouthpiece 120 also includes a second gas flow path that communicates the space in the tobacco pod 130 and the external space of the flavor inhaler 10 so that the user can inhale the tobacco flavor from the tobacco flavor source 132. The second gas flow path has a gas flow outlet, for example, on the mouthpiece end of the mouthpiece 120.

<4. Advantageous Effects>

[0109] According to the method of producing the flavorant-carrying tobacco filler according to the present invention, in the extraction step (S1), the tobacco residue substantially containing no water-soluble component contained in the tobacco material is prepared, and in the subsequent heating step (S2), the amount of carboxyl groups is increased on the surface of the tobacco residue. Therefore, in the subsequent esterification step (S3), the flavorant having the hydroxyl group (hereinafter simply referred to as a flavorant as well) reacts efficiently with many carboxyl groups present on the surface of the tobacco residue without reacting with the carboxylic acid contained in the water-soluble component of the tobacco material, allowing a large amount of flavorant to be carried on the tobacco residue via the ester bond (see Example 1 below).

[0110] In addition, according to the method of producing the flavorant-carrying tobacco filler according to the present invention, during the esterification step (S3), the flavorant is carried on the tobacco residue not only via the ester bond but is also carried on the surface of the tobacco residue by adhesion (see Example 1 below).

[0111] Thus, in the flavorant-carrying tobacco filler according to the present invention, the flavorant is carried on the tobacco residue by both ester bond and adhesion. Because the ester bond is a covalent bond, the flavorant carried by the ester bond is held on the tobacco residue in a chemically stable manner. On the other hand, when the flavorant-carrying tobacco filler according to the present invention is incorporated into the heating type flavor inhaler with the aerosol source being mixed and this heating type flavor inhaler is heated, a transesterification reaction occurs between the flavorant carried by the ester bond and the aerosol source to release the flavorant, and the flavorant carried by adhesion is also released by heating (see Example 2 below).

[0112] Thus, when the flavorant-carrying tobacco filler according to the present invention is incorporated into the heating type flavor inhaler, the flavorant is retained chemically and physically in the tobacco residue without being released during storage (i.e., during non-heating), and the flavorant is released only during inhalation (i.e., during heating). Therefore, the flavorant-carrying tobacco filler according to the present invention is excellent in flavorant storage stability and excellent in flavorant release properties.

[0113] In addition, the transesterification reaction between the flavorant-carrying tobacco filler according to the present invention and the aerosol source generally occurs at a temperature of approximately 100°C to approximately 140°C. The flavorant carried by adhesion is released at a temperature higher than that at which the transesterification reaction occurs. On the other hand, the heating temperature of the heating type flavor inhaler increases gradually after use, and eventually reaches approximately 150°C to approximately 300°C in general.

[0114] The flavorant-carrying tobacco filler according to the present invention can release a sufficient amount of flavorant over a wide range of heating temperatures of the heating type flavor inhaler (see Example 2 below). In particular, the temperature range at which the flavorant-carrying tobacco filler releases the flavorant by the transesterification reaction (approximately 100°C to approximately 140°C) overlaps with a heating temperature range at the initial activation of the heating type flavor inhaler. For this reason, the flavorant-carrying tobacco filler according to the present invention can increase the flavor especially when the heating type flavor inhaler is first activated. Further, in many cases, the heating type flavor inhaler includes the aerosol source having the hydroxyl group, providing a place for the transesterification reaction (flavorant release reaction) of the flavorant-carrying tobacco filler according to the present invention.

Therefore, the flavorant-carrying tobacco filler according to the present invention is particularly suitable as a tobacco filler of a heating type flavor inhaler.

[0115] When the flavorant-carrying tobacco filler according to the present invention is mixed with the aerosol source, the flavorant is considered to be present in the following three states:

(1) eluted in the aerosol source and released into the aerosol source;
(2) carried on the tobacco residue by the ester bond; and
(3) carried on the surface of the tobacco residue by adhesion.

[0116] FIG. 4 schematically shows a condition where the flavorant is present in the above-described three states in the container 131 of the tobacco pod 130. In FIG. 4 and the following description, the flavorant present in the state of (1) is referred to as "flavorant (1)", in the state of (2) as "flavorant (2)", and in the state of (3) as "flavorant (3)". In reality, a large number of flavorant-carrying tobacco fillers are contained in the container 131; however, FIG. 4 shows one enlarged flavorant-carrying tobacco filler contained in the container 131.

[0117] From the results of Example 2 described below, it is considered that when the mixture (tobacco flavor source 132) of the flavorant-carrying tobacco filler and the aerosol source is heated, first, the "flavorant (1)" is released at a temperature lower than approximately 100°C, next, the "flavorant (2)" begins to be released by the transesterification reaction at a temperature of approximately 100°C or more, and then the "flavorant (3)" begins to be released at a temperature of approximately 200°C or more.

[0118] The "flavorant (1)", "flavorant (2)" and "flavorant (3)" are released sequentially as the heating temperature of the heating type flavor inhaler increases, and therefore, the flavorant-carrying tobacco filler according to the present invention can continuously provide the user with the tobacco flavor over the entire inhalation period (see Example 2 below).

[0119] In addition, it is possible to change the timing of releasing the flavorant by changing the abundance ratio of the "flavorant (1)", "flavorant (2)" and "flavorant (3)". For example, a higher abundance ratio of the "flavorant (1)" allows releasing of a greater amount of flavorant before the heating type flavor inhaler reaches an adequate heating temperature. The abundance ratio of the "flavorant (1)" can be increased by not performing the washing step (S4) or by reducing the degree of washing in the washing step (S4) when the flavorant-carrying tobacco filler is produced.

[0120] Alternatively, it is also possible to change the timing of releasing the flavorant by changing the heating temperature or heating time (changing the degree of carbonization) in the heating step (S2) when the flavorant-carrying tobacco filler is produced. For example, if the heating temperature is raised or the heating time is extended in the heating step (S2) when the flavorant-carrying tobacco filler is produced, the resulting flavorant-carrying tobacco filler cannot release the flavorant unless it is heated at a higher temperature, and this can delay the flavorant release timing.

[0121] Alternatively, it is also possible to change the timing of releasing the flavorant by changing the amount of the aerosol source with respect to the flavorant-carrying tobacco filler in the tobacco pod of the heating type flavor inhaler. Since the transesterification reaction that occurs during the flavorant release is a reversible reaction, as the amount of aerosol source is higher, the transesterification reaction is promoted in a flavorant release direction. Thus, with the higher amount of aerosol source, the flavorant-carrying tobacco filler can release the flavorant even when it is heated at a lower temperature, and this can accelerate the flavorant release timing.

<5. Another Embodiment>

[0122] In the above-described embodiment, the flavorant is carried on the tobacco residue obtained from the tobacco material; however, a substrate for carrying the flavorant is not limited to the tobacco residue, and may be a plant residue derived from another plant material. Specifically, according to a preparation method similar to the case of the tobacco residue, a water-soluble component contained in another plant material is extracted with an aqueous solvent from the plant material to prepare a plant residue, and this may be used as a substrate for carrying the flavorant.

[0123] Examples of another plant material include flavorant materials such as mint, peppermint, spearmint, licorice, and carob bean; woody materials such as cherry blossom tree, white oak, and walnut; and other plant materials containing a large amount of cellulose.

[0124] Therefore, according to another embodiment, there is provided a method of producing a flavorant-carrying filler, the method including:

extracting a water-soluble component contained in a plant material with an aqueous solvent from the plant material to obtain a plant extraction liquid and a plant residue;
heating the plant residue to form a carboxyl group in the plant residue; and
mixing the plant residue having the carboxyl group formed therein with a flavorant having a hydroxyl group to cause an esterification reaction between the carboxyl group and the hydroxyl group, thereby obtaining a flavorant-carrying plant residue.

[0125] This method can be performed in a similar procedure to the above-described embodiment in which the flavorant is carried on the tobacco residue.

[0126] The substrate for carrying the flavorant is not limited to the plant residue, and may be an organic substrate capable of forming a carboxyl group by heating. For the organic substrate, a cellulose-based material may be used, an example of which includes cellulose particles, cellulose fibers, paper, and filter paper.

[0127] Therefore, according to another embodiment, there is provided a method of producing a flavorant-carrying filler, the method including:

heating an organic substrate to form a carboxyl group in the organic substrate; and
mixing the organic substrate having the carboxyl group formed therein with a flavorant having a hydroxyl group to cause an esterification reaction between the carboxyl group and the hydroxyl group, thereby obtaining a flavorant-carrying organic substrate.

[0128] This method can be performed in a similar procedure to the above-described embodiment in which the flavorant is carried on the tobacco residue.

[0129] Thus, when a substrate (plant residue or organic substrate) other than the tobacco residue is used as a substrate for carrying a flavorant, the resulting flavorant-carrying filler may be used as a tobacco filler of a heating type flavor inhaler in combination with the "cut tobacco which is ready to be incorporated into a tobacco product (hereinafter simply referred to as cut tobacco as well)" as defined herein. This allows the user to taste both the flavor derived from the flavorant carried on the substrate and the tobacco flavor derived from cut tobacco.

[0130] On the other hand, when the tobacco residue is used as a substrate for carrying a flavorant and the above-described separated tobacco extraction liquid is added to the flavorant-carrying tobacco residue, even if not used together with cut tobacco, the resulting flavorant-carrying tobacco filler can provide the user with both the flavor derived from the flavorant carried on the substrate and the tobacco flavor derived from the tobacco residue and the separated tobacco extraction liquid. Of course, in this case, the flavorant-carrying tobacco filler may be combined with the "cut tobacco which is ready to be incorporated into a tobacco product" and used as a tobacco filler of a heating type flavor inhaler. When the flavorant-carrying tobacco filler is used in combination with cut tobacco, the tobacco flavor can be further increased.

<6. Preferred Embodiments>

[0131] The preferred embodiments of the present invention are summarized below.

[0132] As described above, according to an embodiment, a method of producing a flavorant-carrying tobacco filler includes:

(S1) extracting a water-soluble component contained in a tobacco material with an aqueous solvent from the tobacco material to obtain a tobacco extraction liquid and a tobacco residue;
(S2) heating the tobacco residue to form a carboxyl group in the tobacco residue; and
(S3) mixing the tobacco residue having the carboxyl group formed therein with a flavorant having a hydroxyl group to cause an esterification reaction between the carboxyl group and the hydroxyl group, thereby obtaining a flavorant-carrying tobacco residue.

[0133] According to another embodiment, the method according to the above-described embodiment further includes:

(S6) separating and removing the solvent from the tobacco extraction liquid to prepare a separated tobacco extraction liquid; and
(S7) mixing the separated tobacco extraction liquid with the flavorant-carrying tobacco residue to obtain a mixture of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid.

[0134] As described above, according to a preferred embodiment, a method of producing a flavorant-carrying tobacco filler includes:

(S1) extracting a water-soluble component contained in a tobacco material with an aqueous solvent from the tobacco material to obtain a tobacco extraction liquid and a tobacco residue;
(S2) heating the tobacco residue to form a carboxyl group in the tobacco residue;
(S3) mixing the tobacco residue having the carboxyl group formed therein with a flavorant having a hydroxyl group to cause an esterification reaction between the carboxyl group and the hydroxyl group, thereby obtaining a flavorant-carrying tobacco residue;

(S4) washing the flavorant-carrying tobacco residue to wash away an unesterified flavorant; and
(S5) drying the washed flavorant-carrying tobacco residue to obtain a dried flavorant-carrying tobacco residue.

[0135] According to another embodiment, the method according to the above-described preferred embodiment further includes:

(S6) separating and removing the solvent from the tobacco extraction liquid to prepare a separated tobacco extraction liquid;
(S7) mixing the separated tobacco extraction liquid with the dried flavorant-carrying tobacco residue to obtain a mixture of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid; and
(S8) drying the mixture to obtain a dried mixture.

[0136] According to a more preferred embodiment, in any one of the above-described embodiments, the tobacco material is cut tobacco which is ready to be incorporated into a tobacco product.

[0137] According to a more preferred embodiment, in any one of the above-described embodiments, the tobacco material is a pulverized product of cut tobacco which is ready to be incorporated into a tobacco product.

[0138] According to a more preferred embodiment, in any one of the above-described embodiments, the aqueous solvent is water or water-containing ethanol, preferably water, more preferably water of 20 to 70°C.

[0139] According to a more preferred embodiment, in any one of the above-described embodiments, the extraction (S1) is performed by immersing the tobacco material in warm water at 40 to 60°C for 30 to 180 minutes, or by shaking the tobacco material in warm water at 40 to 60°C for 30 to 180 minutes.

[0140] According to a more preferred embodiment, in any one of the above-described embodiments, the method further includes: separating the tobacco extraction liquid and the tobacco residue after the extraction (S1).

[0141] According to a more preferred embodiment, in any one of the above-described embodiments, the method further includes: separating the tobacco extraction liquid and the tobacco residue after the extraction (S1), and drying the separated tobacco residue.

[0142] According to a more preferred embodiment, in any one of the above-described embodiments, the method further includes: separating the tobacco extraction liquid and the tobacco residue after the extraction (S1), and molding the separated tobacco residue to prepare a molding.

[0143] According to a more preferred embodiment, in any one of the above-described embodiments, the method further includes: separating the tobacco extraction liquid and the tobacco residue after the extraction (S1), and molding the separated tobacco residue to prepare a sheet-like molding.

[0144] According to a more preferred embodiment, in any one of the above-described embodiments, the method further includes: extracting an organic solvent-soluble component contained in the tobacco material with an organic solvent from the tobacco material before the extraction (1).

[0145] According to a more preferred embodiment, in any one of the above-described embodiments, the heating (S2) is performed by heating the tobacco residue at a temperature of 150 to 300°C, preferably 170 to 300°C, more preferably 170 to 270°C, still more preferably 190 to 250°C, and most preferably 230°C.

[0146] According to a more preferred embodiment, in any one of the above-described embodiments, the heating (S2) is performed for 0.5 to 6 hours, preferably 1 to 3 hours.

[0147] According to a more preferred embodiment, in any one of the above-described embodiments, the heating (S2) is performed under a sealed condition.

[0148] According to a more preferred embodiment, in any one of the above-described embodiments, the method further includes: cooling the tobacco residue having the carboxyl group formed therein to a temperature lower than a temperature of the esterification reaction (S3) after the heating (S2).

[0149] According to a more preferred embodiment, in any one of the above-described embodiments, the esterification reaction (S3) is performed by reacting the tobacco residue and the flavorant under an acidic condition at a temperature of 80 to 140°C, preferably 100 to 120°C.

[0150] According to a more preferred embodiment, in any one of the above-described embodiments, the esterification reaction (S3) is performed by reacting the tobacco residue and the flavorant at a pH of 2 or below, preferably a pH of 0.5 to 1.5, at a temperature of 80 to 140°C, preferably 100 to 120°C.

[0151] According to a more preferred embodiment, in any one of the above-described embodiments, the esterification reaction (S3) is performed for 0.5 to 3 hours, preferably 1 to 2 hours.

[0152] According to a more preferred embodiment, in any one of the above-described embodiments, the flavorant is an alcoholic flavorant.

[0153] According to a more preferred embodiment, in any one of the above-described embodiments, the flavorant is menthol, benzyl alcohol, phenethyl alcohol, ethanol, hexanol, hexenol, linalool, coniferyl alcohol, sinapyl alcohol, cinnamyl alcohol, or homovanillyl alcohol.

[0154] According to a more preferred embodiment, in any one of the above-described embodiments, the flavorant is an extract or essential oil, such as peppermint oil, spearmint oil, a licorice extract, a coffee extract, or a cinnamon extract.

[0155] According to a more preferred embodiment, in any one of the above-described embodiments, the esterification reaction (S3) is performed in a sulfuric acid aqueous solution.

[0156] According to a more preferred embodiment, in any one of the above-described embodiments, the method further includes: dividing the reaction mixture into a liquid part and a solid part after the esterification reaction (S3).

[0157] According to a more preferred embodiment, in any one of the above-described embodiments, the method further includes: dividing the reaction mixture into a liquid part and a solid part before the reaction mixture is cooled to 60°C or less, after the esterification reaction (S3).

[0158] According to a more preferred embodiment, in any one of the above-described embodiments, the method further includes: washing the flavorant-carrying tobacco residue after the esterification reaction (S3) to wash away an unesterified flavorant.

[0159] According to a more preferred embodiment, in any one of the above-described embodiments, the washing (S4) is performed by pouring an organic solvent, such as ethanol, over the flavorant-carrying tobacco residue.

[0160] According to a more preferred embodiment, in any one of the above-described embodiments, the washing (S4) is performed by immersing the flavorant-carrying tobacco residue in an organic solvent, such as ethanol, or shaking the flavorant-carrying tobacco residue in an organic solvent, such as ethanol.

[0161] According to a more preferred embodiment, in any one of the above-described embodiments, the washing (S4) is performed after the flavorant-carrying tobacco residue is cooled to 60°C or less, preferably 15 to 25°C.

[0162] According to a more preferred embodiment, in any one of the above-described embodiments, the method further includes: drying the flavorant-carrying tobacco residue after the esterification reaction (S3) or the washing (S4) to obtain a dried flavorant-carrying tobacco residue.

[0163] According to a more preferred embodiment, in any one of the above-described embodiments, the drying (S5) is performed by natural drying or vacuum drying.

[0164] According to a more preferred embodiment, in any one of the above-described embodiments, the separating and removing (S6) is performed by vacuum distillation.

[0165] According to a more preferred embodiment, in any one of the above-described embodiments, the separated tobacco extraction liquid and the flavorant-carrying tobacco residue used in the mixing (S7) are derived from the tobacco extraction liquid and the tobacco residue, respectively, obtained in the same extraction step (S1).

[0166] According to a more preferred embodiment, in any one of the above-described embodiments, the drying (S8) is performed by vacuum drying, freeze drying, or heat drying at a temperature of 50 to 70°C for 0.5 to 2 hours.

[0167] As described above, according to the second aspect, there is provided a flavorant-carrying tobacco filler obtainable by the method according to any one of the above-described embodiments.

[0168] According to another embodiment, the flavorant-carrying tobacco filler comprises:

a tobacco residue; and
a flavorant carried on the tobacco residue by an ester bond between the carboxyl group of the tobacco residue and the hydroxyl group of the flavorant.

[0169] According to a preferred embodiment, the flavorant-carrying tobacco filler comprises:

a tobacco residue;
a flavorant carried on the tobacco residue by an ester bond between the carboxyl group of the tobacco residue and the hydroxyl group of the flavorant; and
a component contained in a tobacco extraction liquid.

[0170] According to a more preferred embodiment, in the above-described preferred embodiments, the tobacco extraction liquid is a tobacco extraction liquid obtained at the time of water extraction of the tobacco material performed to prepare the tobacco residue.

[0171] According to a more preferred embodiment, in any one of the above-described embodiments, the component contained in the tobacco extraction liquid is a water-soluble component of the tobacco material.

[0172] According to a more preferred embodiment, in the above-described preferred embodiments, the component contained in the tobacco extraction liquid is a separated tobacco extraction liquid obtained by extracting the tobacco material with the aqueous solvent and separating and removing the solvent from the tobacco extraction liquid, or a dried separated tobacco extraction liquid obtained by drying the separated tobacco extraction liquid.

[0173] According to a more preferred embodiment, in the above-described preferred embodiments, the flavorant is carried on the tobacco residue in an amount of 0.01 mg or more with respect to 1 g of the tobacco residue.

[0174] According to a more preferred embodiment, in the above-described preferred embodiments, the flavorant is

carried on the tobacco residue in an amount of 0.01 to 50 mg with respect to 1 g of the tobacco residue.

[0175] As described above, according to the third aspect, there is provided a heating type flavor inhaler, comprising a tobacco flavor source,
the tobacco flavor source including:

the flavorant-carrying tobacco filler according to any one of the above-described embodiments; and
an aerosol source mixed with the flavorant-carrying tobacco filler.

[0176] According to a preferred embodiment, in the above-described heating type flavor inhaler, the aerosol source is propylene glycol, glycerin, or a mixture thereof.

[0177] According to a more preferred embodiment, in the above-described preferred embodiments, the aerosol source is contained in an amount of 50 mg or more per 1 g of the flavorant-carrying tobacco filler.

[0178] According to a more preferred embodiment, in the above-described preferred embodiments, the aerosol source is contained in an amount of 50 to 2000 mg per 1 g of the flavorant-carrying tobacco filler.

[0179] According to a more preferred embodiment, in the above-described preferred embodiments, the heating type flavor inhaler comprises normal cut tobacco on which a flavorant is not carried, in addition to the flavorant-carrying tobacco filler according to any one of the above-described embodiments, as a tobacco filler. In this embodiment, the heating type flavor inhaler comprises the flavorant-carrying tobacco filler in an amount of preferably 1 to 99% by mass of the entire tobacco filler contained in the heating type flavor inhaler.

[0180] According to a more preferred embodiment, in the above-described preferred embodiments, the heating type flavor inhaler comprises only the flavorant-carrying tobacco filler according to any one of the above-described embodiments as a tobacco filler.

[0181] According to a more preferred embodiment, in the above-described preferred embodiments, the heating type flavor inhaler is a carbonaceous heat source type inhalation article that heats a mixture of the flavorant-carrying tobacco filler according to any one of the above-described embodiments and an aerosol source with combustion heat of a carbon heat source to generate aerosol.

[0182] According to a more preferred embodiment, in the above-described preferred embodiments, the heating type flavor inhaler is an electrical heating type inhalation article provided with an inhaler body containing a mixture of the flavorant-carrying tobacco filler according to any one of the above-described embodiments and an aerosol source, and a heating device for electrically heating the inhaler body, where the inhalation article heats the mixture with electrical heat to generate aerosol.

[0183] According to a more preferred embodiment, in the above-described preferred embodiments, the heating type flavor inhaler is an electrical heating type inhalation article provided with a refill type tobacco pod containing a mixture of the flavorant-carrying tobacco filler according to any one of the above-described embodiments and an aerosol source, and an inhaler body that heats the tobacco pod with electrical heat to generate aerosol.

[Examples]

[Example 1] Measurement of Amount of Flavorant Carried

[0184] In Example 1, for the flavorant-carrying tobacco filler, the amount of flavorant carried was examined.

1-1. Preparation of Materials

(1) Preparation of Flavorant-carrying Tobacco Filler 1

Extraction step (S1)

[0185] A midrib of flue-cured tobacco was pulverized and used as a "tobacco material". The cut pieces of the midrib of flue-cured tobacco (10 g) were pulverized to a size of 250 μm or less by a pulverizer, and 200 mL of water at 60°C was added, followed by shaking (200 rpm, 2 hours). In this manner, the water-soluble component contained in the midrib was extracted. Subsequently, solid-liquid separation was carried out by filtering. Thereby, the tobacco extraction liquid and the tobacco residue were obtained. The resulting tobacco residue was molded into a sheet with a thickness of approximately 0.1 mm using the paper-making technique. The resulting tobacco molding was cut into a size of 20 mm x 20 mm to obtain cut pieces. This was used as a substrate for carrying the flavorant. In the following description, the cut pieces of the tobacco molding are referred to as a "substrate".

Heating step (S2)

[0186] 0.2 g of the "substrate" obtained in the extraction step (S1) was put in a Mighty Vial No. 5 and a cap was closed. Then, it was heated at 230°C for 3 hours in an aluminum heat-block thermostatic bath. After heating, the sample was cooled to room temperature (25°C). Thereby, the carboxyl group was formed on the substrate.

Esterification step (S3)

[0187] Menthol was used as the "flavorant having a hydroxyl group". In 250 mg of the "substrate having the carboxyl group formed therein" obtained in the heating step (S2), 2.5 g of menthol and 125 μL of 1M sulfuric acid aqueous solution were added, and the mixture was heated at 100°C for 1 hour. This caused an esterification reaction between the carboxyl group of the substrate and the hydroxyl group of menthol, thereby obtaining the "flavorant-carrying substrate".

Washing step (S4)

[0188] The reaction mixture of the esterification reaction was cooled to around 60°C, and a liquid part (unreacted menthol liquid) was removed from the reaction mixture to separate the "flavorant-carrying substrate". Thereafter, the "flavorant-carrying substrate" was cooled back to room temperature. Subsequently, 10 mL of ethanol at room temperature (25°C) was added to the "flavorant-carrying substrate", and shaking extraction (30 minutes, 200 rpm) was performed. Thereafter, solid-liquid separation was performed by centrifugation (15 min, 3000 rmp) to remove the supernatant. The same procedure was repeated three times, and the extra flavorant was washed away.

Drying step (S5)

[0189] After the washing step (S4), the "flavorant-carrying substrate" was dried using a centrifugal evaporator under reduced pressure at 40°C or below for 3 hours, and then air-dried by allowing it to stand for 1 day under the conditions of a temperature of 22°C and a humidity of 63%. In this manner, the "flavorant-carrying tobacco filler 1" was obtained.

(2) Preparation of Flavorant-carrying Tobacco Filler 2 (Comparative Example)

[0190] A "flavorant-carrying tobacco filler 2" was prepared according to the method similar to the method of producing the "flavorant-carrying tobacco filler 1", except that menthol was attached to the "substrate" instead of performing the heating step (S2) and the esterification step (S3). Specifically, menthol was attached by adding menthol to 250 mg of the "substrate" obtained in the extraction step (S1) and heating the mixture at 60°C so that the substrate and menthol were well mixed. Thereafter, the washing step (S4) and the drying step (S5) were performed as described above.

(3) Preparation of Flavorant-carrying Tobacco Filler 3 (Comparative Example)

[0191] A "flavorant-carrying tobacco filler 3" was prepared according to the method similar to the method of producing the "flavorant-carrying tobacco filler 1", except that the heating step (S2) was omitted. Specifically, in 250 mg of the "substrate" obtained in the extraction step (S1), as described in the esterification step (S3), menthol and the sulfuric acid aqueous solution were added and heated at 100°C for 1 hour, thereby performing the esterification step (S3).

1-2. Method

[0192] For the flavorant-carrying tobacco fillers 1 to 3, the amount of flavorant carried was measured.

(1) Measurement of Flavorant bonded to Substrate by adhesion

[0193] 20 μg of the "flavorant-carrying tobacco filler" was weighed in a Mighty Vial (No. 5), and 10 mL of ethanol containing 0.01% of quinoline as an internal standard substance was added, followed by shaking at 200 rpm for 60 minutes. After standing, 0.5 mL of the supernatant was put in GC Vial, and quantitative analysis was conducted by gas chromatography-mass spectrometry (GCMS).

[0194] Here, menthol bonded to the substrate surface by adhesion is eluted in the ethanol solvent, while menthol bonded to the substrate surface via an ester bond is carried on the substrate as it is. Thus, the amount of menthol measured here is the amount of "menthol bonded to the substrate by adhesion" (hereinafter, the menthol amount measured here is referred to "quantitative value 1").

(2) Measurement of Flavorant bonded to Substrate by Ester Bond

[0195] To the remaining of the supernatant used as the sample for GCMS analyses in the measurement of (1), 0.05 mL of 8M sulfuric acid aqueous solution was added, and the mixture was heated at 100°C for 60 minutes. After standing, 0.5 mL of the supernatant was transferred to a GC Vial, and the amount of menthol was measured by GCMS.

[0196] Here, "menthol bonded to the substrate by adhesion" is present in the ethanol solvent, and "menthol bonded to the substrate by ester bond" is also released in the ethanol solvent by the transesterification reaction with ethanol. That is, the menthol amount measured here is the total amount of "menthol bonded to the substrate by adhesion" and "menthol bonded to the substrate by ester bond" (hereinafter, the menthol amount measured here is referred to as "quantitative value 2").

[0197] From the above, the amount of "menthol bonded to the substrate by ester bond" is obtained by the following equation.

Equation: Amount of "menthol bonded to substrate by ester bond" = (Quantitative value 2) - (Quantitative value 1)

1-3. Results

[0198] FIG. 5 shows the measurement results of the amount of flavorant carried. In FIG. 5, the amount of menthol is indicated by the weight of menthol (unit: mg) per 1 g of the substrate for carrying menthol (i.e., the "tobacco residue molding" obtained in the extraction step (S1)).

[0199] The flavorant-carrying tobacco filler 1 had the "menthol bonded to the substrate by adhesion" in an amount equivalent to that of the flavorant-carrying tobacco filler 2. This result indicates that the amount of flavorant bonded by adhesion to the substrate (the tobacco residue molding) is constant regardless of whether the heating step (S2) and the esterification step (S3) are performed.

[0200] In addition, the flavorant-carrying tobacco filler 1 had the "menthol bonded to the substrate by ester bond" in an amount much larger than that of the flavorant-carrying tobacco filler 3. This result indicates the following. In the flavorant-carrying tobacco filler 1, in the heating step (S2), the amount of carboxyl groups increased on the cell wall surfaces of the cells constituting the substrate (the tobacco residue molding), and in the subsequent esterification step (S3), the ester bond occurred between the increased carboxyl groups and hydroxyl groups of the flavorant, making it possible to carry a larger amount of flavorant on the substrate via the ester bond. On the other hand, in the flavorant-carrying tobacco filler 3, because the heating step (S2) was not performed, the amount of carboxyl groups could not be increased on the cell wall surfaces of the cells constituting the substrate, and in the esterification step (S3), only a small amount of flavorant was carried on the substrate via the ester bond.

[0201] Therefore, according to the method according to the present invention, in the heating step (S2), the amount of carboxyl groups can be increased on the cell wall surfaces of the cells constituting the tobacco residue, and in the subsequent esterification step (S3), many flavorants can be carried on the tobacco residue via increased carboxyl groups.

[Example 2] Sensory Evaluation

[0202] In Example 2, flavorant release was checked through sensory evaluation.

2-1. Method

[0203] 5 mg of the "flavorant-carrying tobacco filler 1" was mixed with 5 mg of the mixture of propylene glycol and glycerin (weight ratio, 1:1) to prepare a "tobacco flavor source 1". Similarly, 5 mg of the "flavorant-carrying tobacco filler 3" was mixed with 5 mg of the mixture of propylene glycol and glycerin (weight ratio, 1:1) to prepare a "tobacco flavor source 3".

[0204] Each of the tobacco flavor source 1 and the tobacco flavor source 3 was heated using the thermogravimetry-differential thermal analyzer (TG-DTA) under the following conditions:

Temperature range: 50 to 500°C
Heating rate: 50°C/min
Atmosphere: Air
Sensory evaluation was conducted by evaluating the changes in the quality of scent during heating by sniffing the gas exhausted from the exhaust portion of the thermogravimetry-differential thermal analyzer.

2-2. Results

[0205] In the sensory evaluation (sniffing) of the tobacco flavor source 1, a menthol-derived cooling sensation was provided in all temperature zones measured. In particular, the temperature zone of 150 to 250°C provided a strong menthol-derived cooling sensation.

[0206] This result shows that the flavorant-carrying tobacco filler 1 can continuously release the flavorant over all of the heating temperatures in the presence of the aerosol source, and can release a large amount of flavorant particularly in the heating temperature zone of the heating type flavor inhaler (150 to 250°C), and therefore is useful as a tobacco filler of a heating type flavor inhaler.

[0207] It is considered that the reason why the flavorant-carrying tobacco filler 1 was able to continuously release the flavorant over the entire heating temperature is that the flavorant is present in the three states shown in FIG. 4, i.e., a released state (flavorant (1)), an ester-bond state (flavorant (2)), and an adhering state (flavorant (3)), and the flavorants (1), (2) and (3) are released in this order from a temperature of less than 100°C to a temperature of 300°C.

[0208] On the other hand, in the sensory evaluation (sniffing) of the tobacco flavor source 3, the temperature zone around 100°C provided a strong menthol-derived cooling sensation. When the temperature exceeded 100°C and the menthol-derived cooling sensation was no longer provided, a burnt sweet smell was provided, when the temperature exceeded 200°C, an irritating smell was provided, and when the temperature exceeded 300°C, a strong irritating smell causing nose pain was provided.

[0209] This result shows that the flavorant-carrying tobacco filler 3 released the flavorant very little in the heating temperature zone of the heating type flavor inhaler (150 to 250°C) in the presence of the aerosol source and also released the unpleasant smell, and is therefore not suitable for use as a tobacco filler of a heating type flavor inhaler.

[Example 3] Surface Analysis by Infrared Spectroscopy

[0210] In Example 3, bonding of the flavorant via an ester bond to the carboxyl group of the tobacco residue was checked by surface analysis using infrared spectroscopy.

3-1. Method

[0211] The flavorant-carrying tobacco filler 1 was subjected to the surface analysis by Fourier transform infrared spectroscopy (FT-IR).

[0212] Furthermore, as described in the "1-1. Preparation of Flavorant-carrying Tobacco Filler 1" section of Example 1, "extraction step (S1)" was performed to prepare a "substrate for carrying a flavorant". In addition, as described in the "1-1. Preparation of Flavorant-carrying Tobacco Filler 1" section of Example 1, "extraction step (S1)" and "heating step (S2)" were performed to prepare a "substrate having a carboxyl group formed therein".

[0213] The "substrate for carrying a flavorant" and the "substrate having a carboxyl group formed therein" were subjected to the surface analysis by Fourier transform infrared spectroscopy (FT-IR).

3-2. Results

[0214] FIG. 6 shows the results of analysis by Fourier transform infrared spectroscopy (FT-IR). Three spectra shown in FIG. 6 are, in order from the top, spectrum A of the "substrate for carrying a flavorant", spectrum B of the "substrate having a carboxyl group formed therein", and spectrum C of the "flavorant-carrying tobacco filler 1".

[0215] In spectrum B, an absorption in the vicinity of 1600 [cm^{-1}] shifted to the low wave number side in comparison to spectrum A. This indicates that a carboxyl group was formed in the substrate (the tobacco residue molding). Further, in spectrum B, an absorption in the vicinity of 1600 [cm^{-1}] was broadened in comparison to spectrum A. This indicates that the bonding mode was diversified (conjugation occurred) at a close position linked to carbonyl.

[0216] In spectrum C, an absorption in the vicinity of 1600 [cm^{-1}] shifted to the high wave number side in comparison to spectrum B. This indicates that the flavorant was ester-bonded to the formed carboxyl group.

[0217] These results show that the carboxyl group was formed by the heating step (S2), and that the flavorant was ester-bonded to the formed carboxyl group by the esterification step (S3).

[Example 4] Influence of Heating Conditions of Heating Step (S2) on Amount of Flavorant Carried

[0218] In Example 4, examination was conducted on the influence of the heating conditions of the heating step (S2) on the amount of flavorant carried.

4-1. Method

[0219] The flavorant-carrying tobacco fillers (Samples Nos. 1 to 9) were prepared according to a method similar to the method of preparing the "flavorant-carrying tobacco filler 1" of Example 1, except that the heating temperature and heating time of the heating step (S2) were changed as follows.

[0220] Sample numbers and heating conditions are shown below.

Sample No. 1: 150°C, for 1 hour
Sample No. 2: 150°C, for 3 hours
Sample No. 3: 170°C, for 1 hour
Sample No. 4: 170°C, for 3 hours
Sample No. 5: 190°C, for 1 hour
Sample No. 6: 190°C, for 3 hours
Sample No. 7: 230°C, for 1 hour
Sample No. 8: 230°C, for 3 hours
Sample No. 9: 270°C, for 1 hour

[0221] The amount of "menthol bonded to the substrate by adhesion" and the amount of "menthol bonded to the substrate by ester bond" were measured as described in Example 1.

4-2. Results

[0222] FIG. 7 shows the results of the measurement of the amount of flavorant carried. In FIG. 7, the amount of menthol is indicated by the weight of menthol (unit: mg) per 1 g of the substrate for carrying menthol (i.e., "the tobacco residue molding" obtained in the extraction step (S1)).

[0223] For all of Sample Nos. 1 to 9, the flavorant was bonded to the substrate (the tobacco residue molding) by ester bond, and also bonded to the substrate (the tobacco residue molding) by physical adsorption. Sample Nos. 4 to 9, particularly Sample Nos. 6 to 8, had a large amount of "menthol bonded to the substrate by ester bond".

[0224] These results show that the heating temperature can be set to, for example, 150 to 300°C, preferably 170 to 300°C, more preferably 170 to 270°C, still more preferably 190 to 250°C, and most preferably 230°C, while the heating time can be set to, for example, 1 to 3 hours.

Claims

1. A method of producing a flavorant-carrying tobacco filler, the method comprising:

   extracting a water-soluble component contained in a tobacco material with an aqueous solvent from the tobacco material to obtain a tobacco extraction liquid and a tobacco residue;
   heating the tobacco residue to form a carboxyl group in the tobacco residue; and
   mixing the tobacco residue having the carboxyl group formed therein with a flavorant having a hydroxyl group to cause an esterification reaction between the carboxyl group and the hydroxyl group, thereby obtaining a flavorant-carrying tobacco residue.

2. The method according to claim 1, further comprising:

   separating and removing the solvent from the tobacco extraction liquid to prepare a separated tobacco extraction liquid; and
   mixing the separated tobacco extraction liquid with the flavorant-carrying tobacco residue to obtain a mixture of the flavorant-carrying tobacco residue and the separated tobacco extraction liquid.

3. The method according to claim 1 or 2, wherein the heating is performed by heating the tobacco residue at a temperature of 150 to 300°C.

4. The method according to claim 3, wherein the heating is performed for 0.5 to 6 hours.

5. The method according to any one of claims 1 to 4, wherein the esterification reaction is performed by reacting the tobacco residue and the flavorant under an acidic condition at a temperature of 80 to 140°C.

6. The method according to claim 5, wherein the esterification reaction is performed for 0.5 to 3 hours.

7. The method according to any one of claims 1 to 6, further comprising washing the flavorant-carrying tobacco residue after the esterification reaction to wash away an unesterified flavorant.

8. A flavorant-carrying tobacco filler obtainable by the method according to any one of claims 1 to 7.

9. A flavorant-carrying tobacco filler, comprising:

a tobacco residue; and
a flavorant carried on the tobacco residue by an ester bond between a carboxyl group of the tobacco residue and a hydroxyl group of the flavorant.

10. The flavorant-carrying tobacco filler according to claim 8 or 9, wherein the flavorant is carried on the tobacco residue in an amount of 0.01 mg or more with respect to 1 g of the tobacco residue.

11. A heating type flavor inhaler, comprising a tobacco flavor source,
the tobacco flavor source including:

the flavorant-carrying tobacco filler according to any one of claims 8 to 10; and
an aerosol source mixed with the flavorant-carrying tobacco filler.

12. The heating type flavor inhaler according to claim 11, wherein the aerosol source is contained in an amount of 50 mg or more per 1 g of the flavorant-carrying tobacco filler.

13. A method of producing a flavorant-carrying filler, the method comprising:

heating an organic substrate to form a carboxyl group in the organic substrate; and
mixing the organic substrate having the carboxyl group formed therein with a flavorant having a hydroxyl group to cause an esterification reaction between the carboxyl group and the hydroxyl group, thereby obtaining a flavorant-carrying organic substrate.

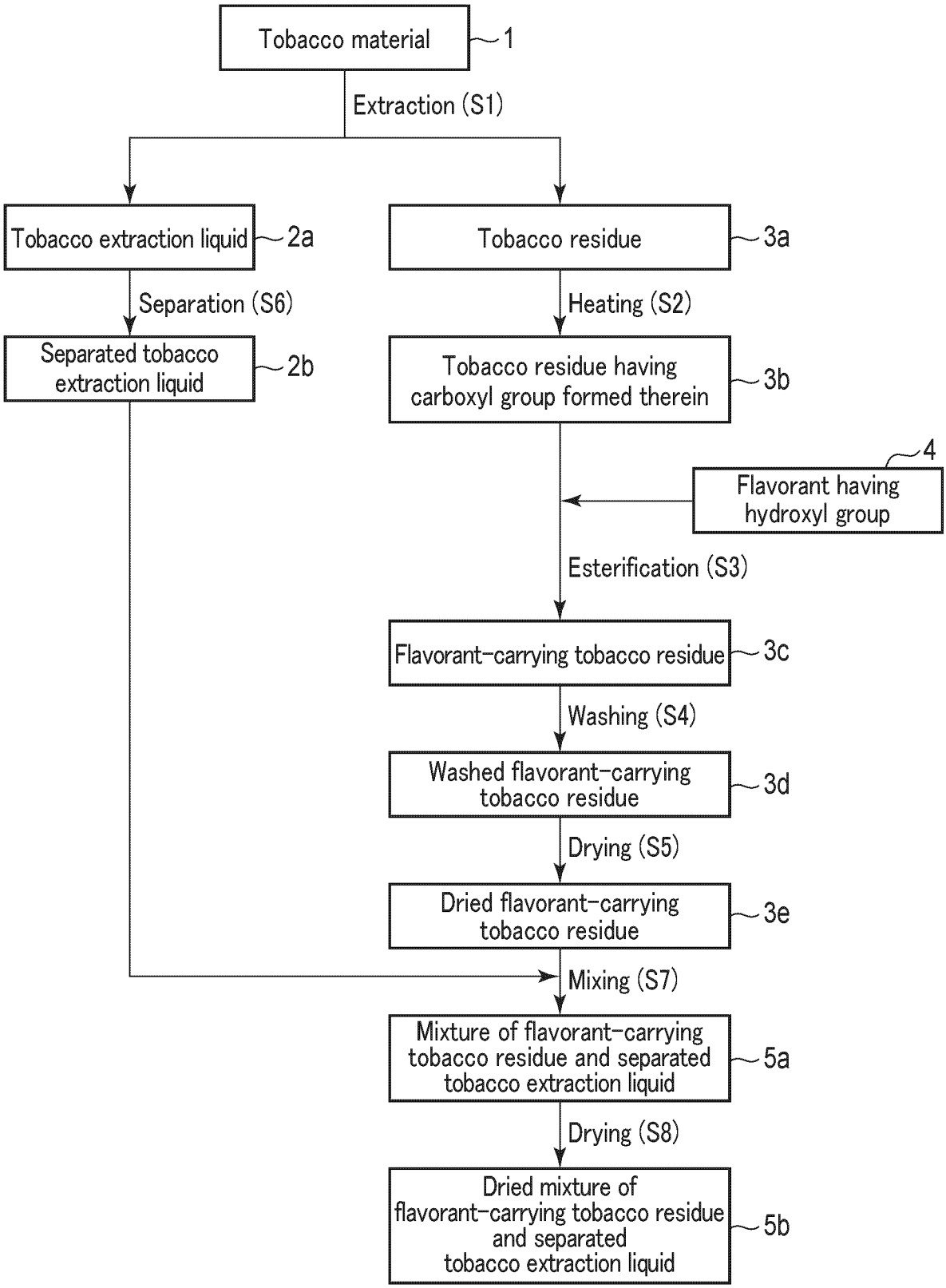
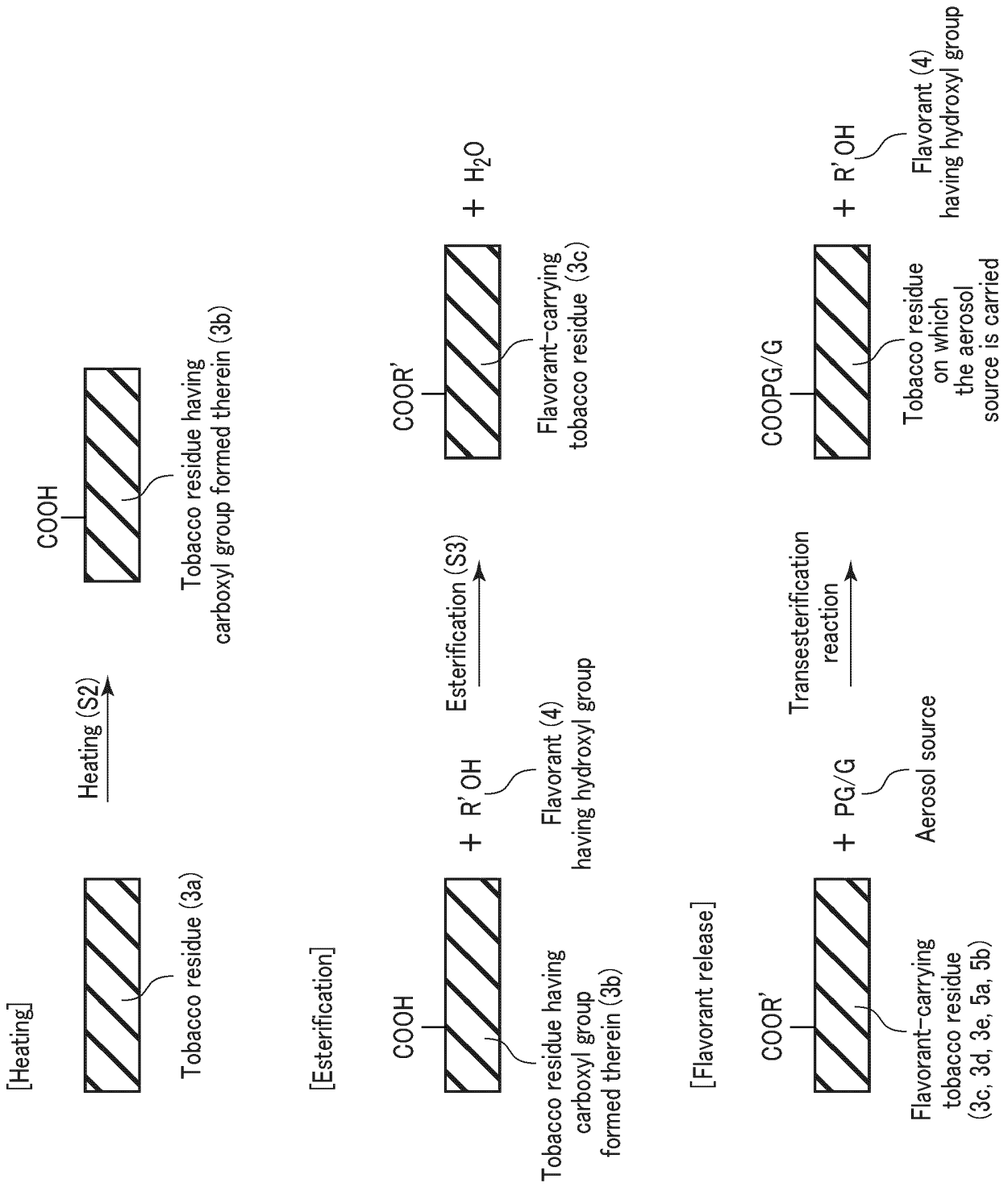

Tobacco material
1
Extraction (S1)
Tobacco extraction liquid
2a
Tobacco residue
3a
Separation (S6)
Heating (S2)
Separated tobacco extraction liquid
2b
Tobacco residue having carboxyl group formed therein
3b
4
Flavorant having hydroxyl group
Esterification (S3)
Flavorant-carrying tobacco residue
3c
Washing (S4)
Washed flavorant-carrying tobacco residue
3d
Drying (S5)
Dried flavorant-carrying tobacco residue
3e
Mixing (S7)
Mixture of flavorant-carrying tobacco residue and separated tobacco extraction liquid
5a
Drying (S8)
Dried mixture of flavorant-carrying tobacco residue and separated tobacco extraction liquid
5b

FIG. 1

FIG. 2


[Heating]
Tobacco residue (3a)
Heating (S2)
COOH
Tobacco residue having carboxyl group formed therein (3b)
[Esterification]
COOH
Tobacco residue having carboxyl group formed therein (3b)
+ R'OH
Flavorant (4) having hydroxyl group
Esterification (S3)
COOR'
Flavorant-carrying tobacco residue (3c)
+ H_2O
[Flavorant release]
COOR'
Flavorant-carrying tobacco residue (3c, 3d, 3e, 5a, 5b)
+ PG/G
Aerosol source
Transesterification reaction
COOPG/G
Tobacco residue on which the aerosol source is carried
+ R'OH
Flavorant (4) having hydroxyl group

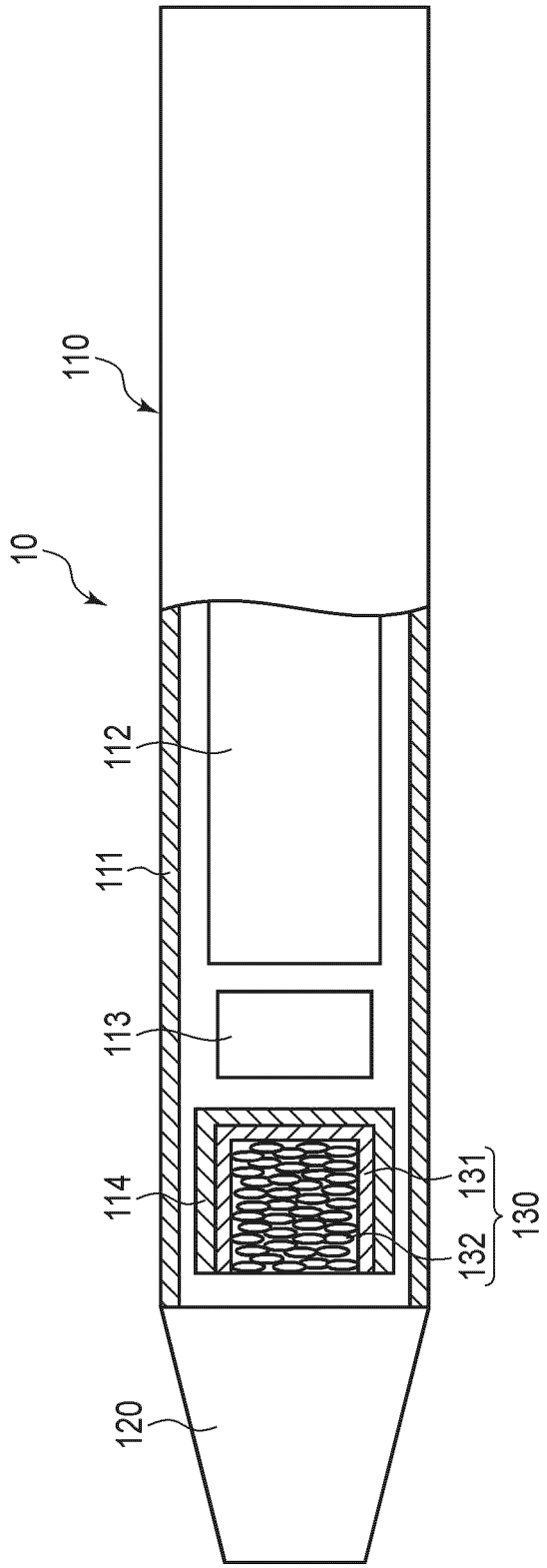


FIG. 3

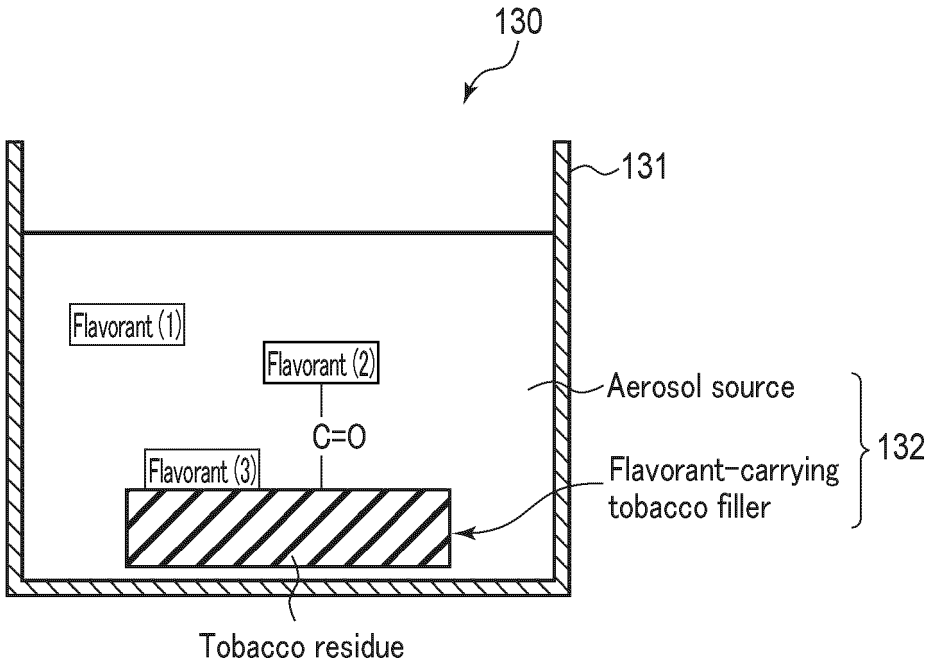

130
131
Flavorant (1)
Flavorant (2)
C=O
Flavorant (3)
Aerosol source
Flavorant-carrying tobacco filler
132
Tobacco residue

FIG. 4

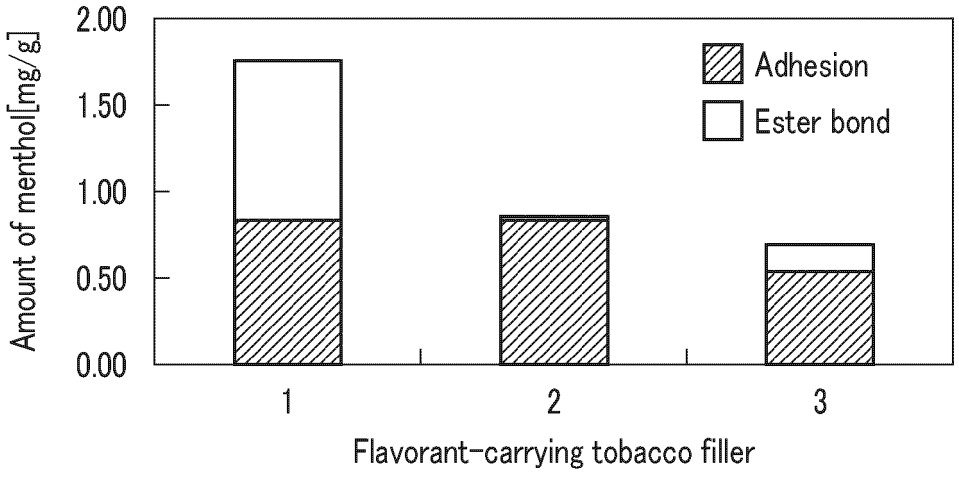

Amount of menthol[mg/g]
2.00
1.50
1.00
0.50
0.00
Adhesion
Ester bond
1
2
3
Flavorant-carrying tobacco filler

FIG. 5

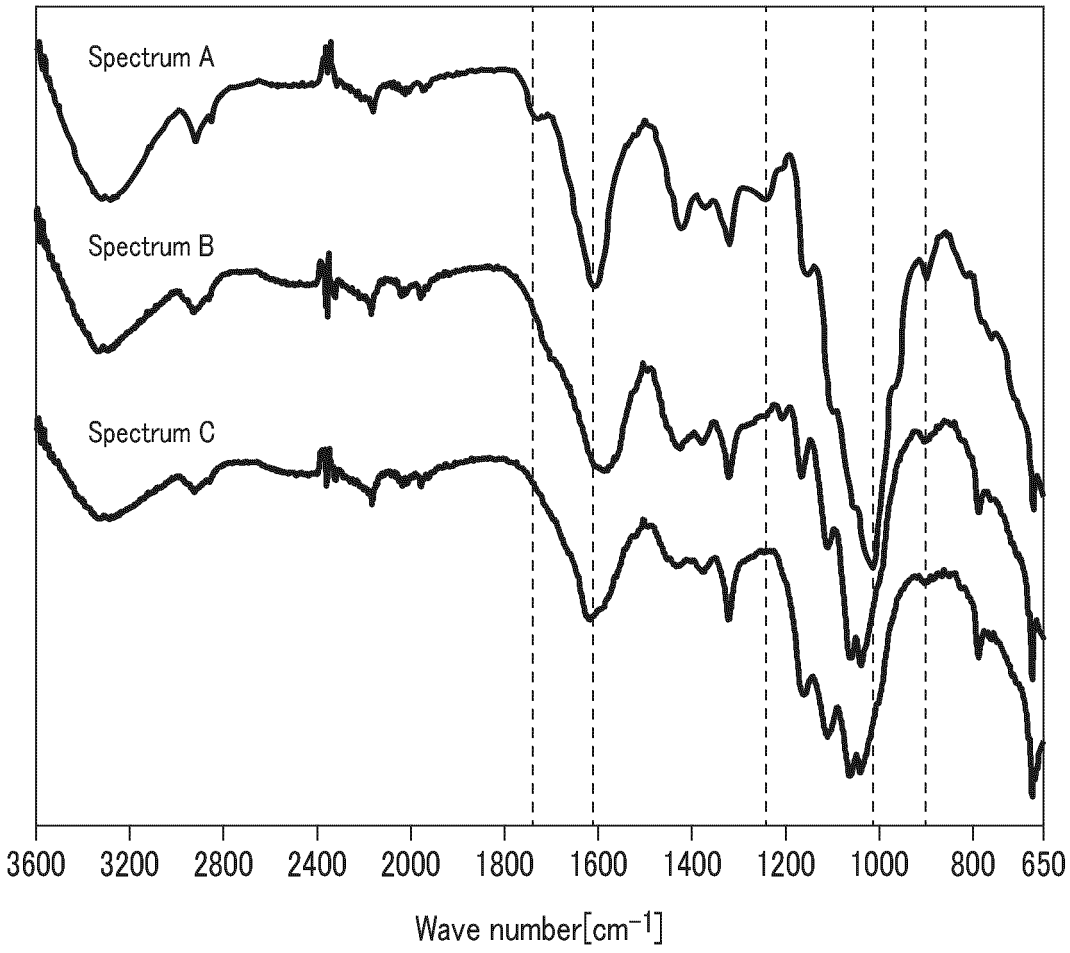

Spectrum A
Spectrum B
Spectrum C
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
650
Wave number[cm^{-1}]

FIG. 6

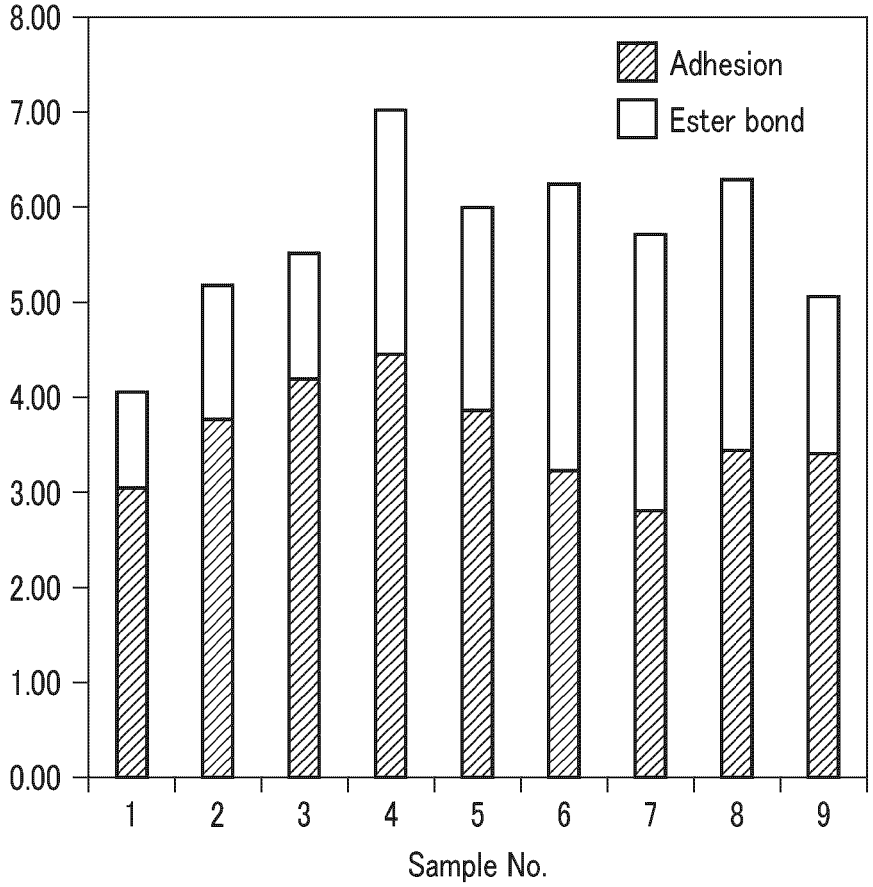

8.00
7.00
6.00
5.00
4.00
3.00
2.00
1.00
0.00
Adhesion
Ester bond
1
2
3
4
5
6
7
8
9
Sample No.

FIG. 7

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2017/041338

A. CLASSIFICATION OF SUBJECT MATTER
Int. Cl. A24B15/30(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)
Int. Cl. A24B15/30, A24B15/24

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Published examined utility model applications of Japan 1922-1996
Published unexamined utility model applications of Japan 1971-2018
Registered utility model specifications of Japan 1996-2018
Published registered utility model applications of Japan 1994-2018

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X A	JP 2010-534071 A (PHILIP MORRIS PRODUCTS S.A.) 04 November 2010, claims 1, 9, paragraph [0005] & US 2009/0028803 A1, claims 1, 13, paragraph [0010] & US 2013/0336901 A1 & WO 2009/013632 A2 & EP 2166877 A	9-12 1-8, 13
A	JP 47-010040 B1 (JAPAN TOBACCO AND SALT PUBLIC CORPORATION) 25 March 1972, entire text, all drawings (Family: none)	1-13
A	WO 2004/098323 A1 (JAPAN TOBACCO INC.) 18 November 2004, entire text, all drawings & US 2006/0065279 A1, entire text, all drawings & EP 1623634 A1	1-13

☐ 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
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"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
08.02.2018	20.02.2018
Name and mailing address of the ISA/ Japan Patent Office 3-4-3, Kasumigaseki, Chiyoda-ku, Tokyo 100-8915, Japan	Authorized officer Telephone No.

Form PCT/ISA/210 (second sheet) (January 2015)

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- WO 2013098920 A **[0003]**
- WO 2006073065 A **[0089]**
- WO 2010110226 A **[0089]**
- WO 2013025921 A **[0089]**