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(54) TOBACCO FILLER FOR NON-COMBUSTION TYPE HEAT SMOKING ARTICLE

(57) The purpose of the present invention is to provide a tobacco filler that can maintain a good smoking flavor and yet reduce "a feeling of lessened smoking flavor" in a non-combustion type heat smoking article served in the form of a tobacco material-containing filler to be heated. Provided is a tobacco filler for a non-combustion type heat smoking article, said tobacco filler containing a tobacco material and an aerosol-forming liquid, wherein the aerosol-forming liquid contains diacetin and/or monoacetin and the acetic acid production rate constant of the tobacco material is $1.25 \times 10^{-8} s^{-1}$ or less.

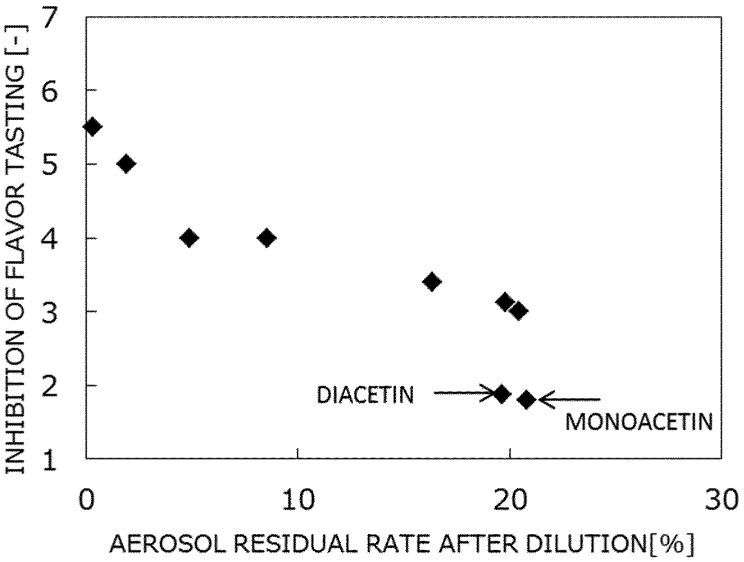

Fig.1

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Description

[Technical Field]

[0001] The present invention relates to a tobacco filler for a non-combustion type heat smoking article that fills a non-combustion type heat smoking article for use.

[Background Art]

[0002] In recent years, non-combustion type heat smoking articles that replace cigarettes and allow tasting of a flavor without combusting tobacco have been developed, and one which is a pod-shaped container containing a flavor component and a component capable of generating aerosol filling the container or one having a heat source at a tip thereof are typically known.

[0003] It has been also reported that glycerol, triethylene glycol and propylene glycol are added to a filler in such non-combustion type heat smoking articles (see Patent Document 1).

[Citation List]

[Patent Document]

[0004] [Patent Document 1] Japanese Patent Application Publication No. S63-148975

[Summary of Invention]

[Technical Problem]

[0005] During smoking with non-combustion type heat smoking articles, it is required to achieve a sufficient evaporation of flavor components and reduce so-called "inhibition of flavor tasting".

[0006] For example, Patent Document 1 described above indicates that flavor tasting is preferable when an aerosol generating liquid used is glycerol, triethylene glycol and propylene glycol. Meanwhile, propylene glycol added in the invention disclosed in Patent Document 1 has low boiling point and high vapor pressure, and thus is liable to volatilize in the oral cavity. Because of this, propylene glycol may create an atmosphere in which components other than propylene glycol contained in aerosol are also liable to volatilize, thereby undergoing phase transition to gas phase as propylene glycol does. The volatilized components contain, in addition to flavor components, substances that inhibit sensing of flavor. Therefore, in order to eliminate inhibition of flavor tasting, it is required to significantly increase the amount of propylene glycol generated *per se* or convert the same to aerosol generating liquid that may hardly be volatilized.

[0007] In view of the above, an object of the present invention is to provide a tobacco filler for a non-combustion type heat smoking article that can maintain preferable flavor tasting and suppress "inhibition of flavor tasting".

[Solution to Problem]

[0008] The inventors of the present invention carried out an extensive study in order to solve the problem and, as a result, found that the above problem can be solved by adding an aerosol generating liquid containing at least one of diacetin and monoacetin to a tobacco filler and using a tobacco material having an acetic acid generation rate constant of 1.25×10^{-8} s^{-1} or less, thereby completing the present invention.

[0009] Thus, the present invention is as follows.

[1] A tobacco filler for a non-combustion type heat smoking article containing a tobacco material and an aerosol generating liquid, wherein:

the aerosol generating liquid contains at least one of diacetin and monoacetin;
the tobacco material has an acetic acid generation rate constant of 1.25×10^{-8} s^{-1} or less and an added amount of the aerosol generating liquid is 50% by weight to 300% by weight relative to the weight of the tobacco material.

[2] The tobacco filler according to [1], wherein the aerosol generating liquid contains both diacetin and monoacetin.
[3] The tobacco filler according to [1] or [2], wherein the aerosol generating liquid contains 50% by weight or more in total of diacetin and monoacetin relative to the aerosol generating liquid.
[4] A pod for a non-combustion type heat smoking article, containing the tobacco filler according to any of [1] to [3]

filling therein.

[5] A non-combustion type heat smoking article, containing the tobacco filler according to any of [1] to [3].

[Effects of Invention]

[0010] According to the present invention, preferable flavor tasting can be maintained while suppressing "inhibition of flavor tasting" in a non-combustion type heat smoking article in which a filler containing a tobacco material is heated.

[Brief Description of Drawings]

[0011]

Fig. 1 is a view illustrating the relationship between an aerosol residual rate after dilution and inhibition of flavor tasting.
Fig. 2 is a view illustrating the relationship between the added amount of diacetin and inhibition of flavor tasting.
Fig. 3 is a view illustrating the relationship between the added amount of water and the acetic acid amount.
Fig. 4 is a view illustrating the relationship between the acetic acid amount and sourness.
Fig. 5 is a view illustrating the relationship between the days of storage and the amount of acetic acid produced.
Fig. 6 is a view illustrating the relationship between the days of storage and the proportion of acetic acid produced.
Fig. 7 is a view illustrating the relationship between the days of storage and $-\ln(1-C/C_{max})$.
Fig. 8 is a section view illustrating an example of a non-combustion type heat smoking article.

[Description of Embodiments]

[0012] The present invention is described by way of specific examples. However, the present invention is not limited to the following content, and may be appropriately modified and practiced without departing from the scope of the present invention.

<Tobacco filler for a non-combustion type heat smoking article>

[0013] The tobacco filler for a non-combustion type heat smoking article according to one embodiment of the present invention (hereinafter sometimes abbreviated as "tobacco filler of the present invention") contains a tobacco material having an acetic acid generation rate constant of 1.25×10^{-8} s^{-1} or less and an aerosol generating liquid containing at least one of diacetin and monoacetin, wherein an added amount of the aerosol generating liquid is 50% by weight to 300% by weight relative to the weight of the tobacco material.

<Acetic acid generation rate constant of the tobacco material>

[0014] The tobacco material included in the tobacco filler of the present invention has an acetic acid generation rate constant of 1.25×10^{-8} s^{-1} or less.

[0015] Because the tobacco material has an acetic acid generation rate constant of 1.25×10^{-8} s^{-1} or less, an activity of a hydrolase (acetyl esterase) in the tobacco material included in the tobacco filler is sufficiently suppressed and the amount of acetic acid generated by hydrolysis of monoacetin and diacetin during storage of the tobacco filler is reduced. As a result, deterioration of flavor tasting during use of the tobacco filler can be prevented.

[0016] The tobacco material more preferably has an acetic acid generation rate constant of 1.17×10^{-8} s^{-1} or less and particularly preferably 1.00×10^{-8} s^{-1} or less.

[0017] From Fig. 6 illustrating the relationship of the proportion of acetic acid generated relative to the days of storage confirmed in Examples described hereinbelow, generation of acetic acid (decomposition of diacetin) behaves according to first-order reaction until day 21 of storage. Based on this, the acetic acid generation rate constant k organized relative to the days t of storage can be represented by the following first-order reaction rate equation.

$$kt = -\ln\left(1 - \frac{C}{C_{max}}\right)$$

In the above equation, C represents the acetic acid amount and C_{max} represents the maximum amount of acetic acid generated by hydrolysis of diacetin added.

[0018] The acetic acid generation rate constant is calculated under the following conditions.

[0019] A mixture of a tobacco material and diacetin is prepared by adding 100 mg of diacetin to 100 mg (wet weight) of the tobacco material.

[0020] The prepared mixture is stored for 2 months under conditions of 22°C and humidity of 60%.

[0021] The mixture after the storage is placed in a screw vial and subjected to extraction by shaking in a methanol solvent for 40 minutes, acetic acid is assayed by GC-MS and the acetic acid generation rate constant is calculated according to the above equation.

[0022] The tobacco material having the above acetic acid generation rate constant may be obtained by subjecting shredded tobacco or sheet-shaped tobacco obtained by grinding leaf tobacco into powder and then shaping the same to a treatment for inactivating hydrolase as described hereinafter.

[0023] The type of leaf tobacco used for the tobacco material is not particularly limited and examples thereof include flue-cured tobacco, Burley tobacco, Japanese domestic tobacco, regenerated tobacco and the like. Examples of the part to be used include the leaf (expanded tobacco), stem, rib (cut stem), root, flower and the like.

[0024] The dimension of the shredded tobacco is not particularly limited. The shredded tobacco may have a sphere equivalent diameter of generally 1.5 mm or less and preferably 0.5 mm or less and generally 0.01 mm or more as measured by a projected area method (such as the method using, for example, Camsizer (Retsch GmbH)).

[0025] When shredded tobacco is used, tobacco leaves may be cut into the size (maximum diameter) of 0.01 mm to 100 mm.

[0026] In the present invention, the tobacco material used is obtained by treating a starting material for obtaining the tobacco material so as to have an acetic acid generation rate constant of 1.25×10^{-8} s^{-1} or less.

[0027] Examples of the treatment include a treatment for inactivating hydrolase that affects the acetic acid generation rate constant and is included in tobacco leaves or the like in a starting material that provides the tobacco material.

[0028] Examples of the treatment for inactivating hydrolase in tobacco leaves include a treatment that causes structural change of a protein that forms the enzyme such as heat treatment. Specifically, an embodiment of heating at a temperature of 130°C or higher for 60 minutes or more may be mentioned. When the temperature is increased, the heating time may be appropriately decreased.

[0029] Another treatment for inactivating hydrolase may be physical treatments such as a treatment of heating simultaneously with drying or freeze-drying.

[0030] Another treatment may be chemical treatments in which a starting material that provides the tobacco material is used in the presence of an organic solvent or in which an acid/base is added to tobacco leaves.

[0031] The tobacco filler of the present invention contains the tobacco material at generally 20% by weight or more, preferably 30% by weight or more and more preferably 40% by weight or more, and generally 80% by weight or less, preferably 70% by weight or less and more preferably 60% by weight or less. The content within the above range can maintain preferable flavor tasting during use and effectively suppress "inhibition of flavor tasting".

[0032] The tobacco filler of the present invention contains an aerosol generating liquid that contains at least one of diacetin and monoacetin.

[0033] As demonstrated in Examples described hereinafter, when the tobacco filler of the present invention contains an aerosol generating liquid that contains at least one of diacetin and monoacetin, aerosol generated during use of a non-combustion type smoking article containing the tobacco filler of the present invention is less volatilized due to dilution in oral cavity, and components that can inhibit sensing of flavor components included in aerosol can be prevented from undergoing phase transition to gas phase. Due to the above, inhibition of flavor tasting can be reduced.

[0034] In the present invention, an extent of volatilization of generated aerosol due to dilution in oral cavity is evaluated by measuring the "aerosol residual rate after dilution". A higher aerosol residual rate after dilution means a lower inhibition of flavor tasting.

[0035] The aerosol generating liquid used in the present invention contains at least one of diacetin and monoacetin.

[0036] As to the content thereof, the added amount of the aerosol generating liquid is 50% by weight to 300% by weight relative to the tobacco material included in the tobacco filler.

[0037] When the content of the aerosol generating liquid that contains at least one of diacetin and monoacetin is 50% by weight or more relative to the tobacco material, inhibition of flavor tasting can be effectively reduced.

[0038] Meanwhile, the upper limit of the content of the aerosol generating liquid that contains at least one of diacetin and monoacetin is 300% by weight or less in order to allow smooth heating of the tobacco filler during use thereof in a non-combustion type smoking article.

[0039] It is difficult to prepare liquids respectively containing solely diacetin and monoacetin. A commercially available "diacetin" solution contains about 42% by weight of diacetin and about 38% by weight of monoacetin. A commercially available "monoacetin" solution contains about 45% by weight of monoacetin and about 36% by weight of diacetin.

[0040] Because of this, when, for example a commercially available "diacetin" solution is added at 50% by weight relative to the tobacco material as a solution that contains at least one of diacetin and monoacetin, the content of diacetin relative to the tobacco material is about 21% by weight and the content of monoacetin is about 19% by weight.

[0041] A commercially available "diacetin" solution contains 81% by weight in total of diacetin and monoacetin, and a commercially available "monoacetin" solution contains 81% by weight in total of diacetin and monoacetin.

[0042] Thus, when a commercially available "diacetin" solution or a commercially available "monoacetin" solution is added at 50% by weight relative to the tobacco material in the tobacco filler of the present invention, the total amount of diacetin and monoacetin relative to the tobacco material is about 40% by weight.

[0043] Embodiments of the aerosol generating liquid used in the present invention include one containing both diacetin and monoacetin.

[0044] The aerosol generating liquid used in the present invention preferably contains diacetin and monoacetin in total at 50% by weight or more, more preferably 70% by weight or more and particularly preferably 75% by weight or more.

[0045] The aerosol generating liquid may optionally contain other components in addition to at least one of diacetin and monoacetin as described above.

[0046] Examples of other components include acids listed hereinafter having a first acid dissociation constant of 4.0 to 6.0 inclusive and a boiling point of 366°C to 600°C. The "first acid dissociation constant" means an acid dissociation constant in water at normal temperature (25°C).

[0047] The "boiling point" means a boiling point at pressure of 760 mmHg.

[0048] Examples of the acid include ascorbic acid, isoascorbic acid, heneicosanoic acid, lignoceric acid, octacosanoic acid, nonadecanoic acid and the like.

[0049] Among these, ascorbic acid, isoascorbic acid and the like are particularly preferred.

[0050] The acid as described above allows easier suppression of a reduction in the amount of evaporated flavor components and more effective suppression of "inhibition of flavor tasting".

[0051] When the tobacco filler of the present invention contains the above acid, the content thereof is generally 0.25% by weight or more and preferably 1% by weight or more and generally 10% by weight or less. The content within the above range allows easier suppression of a reduction in the amount of evaporated flavor components and more effective suppression of "inhibition of flavor tasting".

[0052] Examples of additives other than the above acid include polyhydric alcohols such as glycerol, propylene glycol, triethylene glycol and tetraethylene glycol; and aliphatic esters of carboxylic acids such as methyl stearate, dimethyl dodecanedioate and dimethyl tetradecanedioate.

[0053] The component used is not limited to one type and may be a combination of two or more.

[0054] The tobacco filler of the present invention is expected to have an increase in the acetic acid amount in the tobacco filler, when stored for 2 months or more after preparation, of only 1.1% by weight or less relative to the weight of the aerosol generating liquid added. This is because hydrolase included in the tobacco material is inactivated as described above, and thus the tobacco material has a low acetic acid generation rate constant.

[0055] Accordingly, the tobacco filler of the present invention also has excellent storage stability.

[0056] The tobacco filler of the present invention contains shredded tobacco and an aerosol generating liquid. Generally, components contained in shredded tobacco such as water is dissolved in the aerosol generating liquid, and thus it can be regarded that the tobacco filler of the present invention also contains water.

[0057] The tobacco filler of the present invention contains water at generally 5% by mass or more, preferably 7.5% by mass or more and more preferably 10% by mass or more, and generally 30% by mass or less, preferably 25% by mass or less and more preferably 20% by mass or less of the whole tobacco filler. The content within the above range can maintain preferable flavor tasting during use and allows more effective suppression of "inhibition of flavor tasting".

[0058] The tobacco filler of the present invention may be distributed in the form of being included in a container such as a pod in order to be used for a non-combustion type heat smoking article. The pod on this occasion may have a well-known shape and may be made of any material without limitation including metals having high thermal conductivity such as aluminium.

[0059] The amount of the tobacco filler added to the pod may be appropriately adjusted according to the type of the marketed product.

[0060] The tobacco filler of the present invention is a tobacco filler for a non-combustion type heat smoking article containing shredded tobacco and an aerosol generating liquid. The non-combustion type heat smoking article which is filled with the tobacco filler of the present invention is not particularly limited as to the specific structure and the like thereof, and may appropriately be any well-known non-combustion type heat smoking article. The non-combustion type heat smoking article is hereinafter described by way of specific examples.

[0061] Examples of the non-combustion type heat smoking article include the one that has the structure as a non-combustion type heat smoking article 10 illustrated in Fig. 8. Fig. 8 is a section view of the cylindrical non-combustion type heat smoking article 10 along the longitudinal direction. The non-combustion type heat smoking article 10 has the structure provided with a battery 101, a pod 103 that accommodates a filler 102 and a heater 104 and a mouthpiece 105. By filling the pod 103 with the tobacco filler of the present invention and heating the same, aerosol is generated.

[0062] The heating temperature of the tobacco filler in the non-combustion type heat smoking article is generally 22°C or higher, preferably 100°C or higher and more preferably 150°C or higher, and generally 350°C or lower, preferably 300°C or lower and more preferably 250°C or lower. When the tobacco filler of the present invention is used for a non-combustion type heat smoking article having a heating temperature of a tobacco filler within the above range, properties of the tobacco filler of the present invention can be more effectively utilized in order to solve the problem on "inhibition of flavor tasting".

Examples

[0063] The present invention is more specifically described hereinafter by way of Examples. The present invention may be appropriately modified within the scope of the present invention.

<Examples 1 and 2 and Comparative Examples 1 to 7: Verification of an effect by variation in aerosol generating liquids>

[0064] In order to verify an effect of variation of aerosol generating liquids on a reduction of inhibition of flavor tasting, 100 mg of various aerosol generating liquids indicated in Table 1 were respectively added to 100 mg of shredded flue-cured tobacco (product in Japan, hereinafter also merely referred to as shredded tobacco; acetic acid generation rate constant: $1.17 \times 10^{-8}\ s^{-1}$) as in Example 5 hereinafter as the tobacco material to prepare samples .

[0065] Shredded tobacco used was obtained by grinding tobacco in a household mixer followed by shaking on a sieve (AS 200, manufactured by Retsch GmbH) under the condition: amplitude-1.5 mm/"g" for 2 minutes, and had a mesh size of 0.5 mm or less. The prepared samples were respectively added to a pod dedicated to a product with the product name "Ploom" marketed by Japan Tobacco Inc. so as to be attached to the pod, and stored for 2 days or more under conditions of 22°C and humidity of 60%.

[0066] The "diacetin" used in present Examples is a commercially available solution and contains about 42% by weight of diacetin and about 38% by weight of monoacetin as described above. The "monoacetin" used in present Examples is also a commercially available solution and contains about 45% by weight of monoacetin and about 36% by weight of diacetin as described above.

[0067] It was confirmed that the heating temperature (during stable operation) of shredded tobacco using Ploom was about 160°C to 170°C by preliminary measurement using a thermocouple.

[0068] As described above, the "extent of volatilization of aerosol" that is important for measuring a reduction of inhibition of flavor tasting was evaluated by "aerosol residual rate after dilution" which was a ratio between the aerosol amount before dilution and the aerosol amount after dilution with cleansed air measured. A measurement of the aerosol amount before dilution was conducted on a smoking machine (Borgwaldt, RM-26) by attaching the prepared pod to Ploom and measuring initial 5 puffs under specified smoking conditions (55 ml/2 s, smoking interval: 30 s).

[0069] The aerosol amount after dilution was measured as follows: aerosol generated in the same manner as described above was accumulated in an SUS tube (capacity: about 127 cc, length: 25.0 cm, inner diameter: 2.54 cm) and cleaned air was allowed to pass through the tube to create a pseudo dilution condition, aerosol was then allowed to pass through a charcoal packed bed (100 mg) to remove components existing in a gas phase, and then the aerosol amount for initial 5 puffs was measured with a Cambridge filter. The sensory evaluation of the prepared samples was carried out by four evaluators and "inhibition of flavor tasting" was evaluated on a 7-grade scale of 1 to 7. In the results of the present Examples, when the evaluated score on inhibition of flavor tasting was 2.0 or less, evaluators could sufficiently recognize the difference, and thus the region was regarded to have an excellent effect.

[0070] Fig. 1 indicates the relationship between the aerosol residual rate after dilution and inhibition of flavor tasting when various aerosol generating liquids indicated in Table 1 were used. From Fig. 1, it is found that the inhibition of flavor tasting tends to decrease as the aerosol residual rate after dilution increases. Namely, it is speculated that when generated aerosol is difficult to volatilize due to dilution in oral cavity and the aerosol amount remaining in the oral cavity after dilution therein is high, substances that may inhibit sensing flavor in aerosol is difficult to undergo phase transition to gas phase, thereby reducing inhibition of flavor tasting. It was also found that diacetin had high aerosol residual rate after dilution, and thus was, as a result, an aerosol generating liquid that provided the lowest flavor tasting inhibition.

[Table 1]

[0071]

Table 1 Aerosol generating liquids and evaluation results

Classification	Aerosol generating liquid	Boiling point [°C]	Inhibition of flavor tasting [-]	Aerosol residual rate after dilution [%]
Comparative Example 1	Propylene glycol	188	5.5	0.3

(continued)

Classification	Aerosol generating liquid	Boiling point [°C]	Inhibition of flavor tasting [-]	Aerosol residual rate after dilution [%]
Comparative Example 2	1,3-Butanediol	208	4.0	4.9
Comparative Example 3	1,3-Propanediol	217	5.0	1.9
Example 1	Monoacetin	258	1.8	20.8
Comparative Example 4	Triacetin	259	4.0	8.6
Example 2	Diacetin	261	1.9	19.7
Comparative Example 5	Triethylene glycol	285	3.4	16.3
Comparative Example 6	Glycerol	290	3.0	20.4
Comparative Example 7	Triethyl citrate	294	3.1	19.8

<Examples 2 to 4 and Comparative Examples 8 to 14> Study on the amount that is effective for reduction of inhibition of flavor tasting

[0072] As described above, diacetin and monoacetin are respectively difficult to be isolated, and thus solutions containing diacetin and monoacetin have very similar compositions. Thus, in the following Examples, a solution containing diacetin (commercially available product) was used as a typical example for studying the amount that was effective for reduction of inhibition of flavor tasting.

[0073] In order to study the amount that was effective for reduction of inhibition of flavor tasting, to 100 mg of shredded Burley tobacco (product in Japan), diacetin and, as a comparison, propylene glycol were respectively added at the amounts indicated in Table 2 to prepare samples. The samples were prepared in the same manner as previous Examples and stored for 2 days or more under the conditions of 22°C and humidity of 60%. The sensory evaluation of the prepared samples was carried out by four evaluators and "inhibition of flavor tasting" was evaluated on a 7-grade scale of 1 to 7. In the results of the present Examples, when the evaluated score on inhibition of flavor tasting was 2.0 or less, evaluators could sufficiently recognize the difference, and thus the region was regarded to have an excellent effect.

[0074] Fig. 2 indicates the relationship between the amount of solution added relative to the weight of shredded tobacco and inhibition of flavor tasting. From Fig. 2, it is found that inhibition of flavor tasting was as high as 6.8 when an aerosol generating liquid was not added to shredded tobacco (when the addition of the solution relative to the weight of shredded tobacco was 0% by weight) . Meanwhile, when diacetin was added to shredded tobacco as the aerosol generating liquid, inhibition of flavor tasting decreased as the addition of solution relative to the weight of shredded tobacco increased.

[0075] Further, when the addition of solution relative to the weight of shredded tobacco was 50% by weight or more, inhibition of flavor tasting significantly decreased. Therefore, the amount of diacetin added that is effective for reduction of inhibition of flavor tasting is 50% by weight or more relative to the weight of shredded tobacco. It was found that when propylene glycol, as a comparison, was added as an aerosol generating liquid at 100% by weight relative to the weight of shredded tobacco, inhibition of flavor tasting was at a similar level as no aerosol generating liquid was added to shredded tobacco. From this result, it is found that for reduction of inhibition of flavor tasting, not only an increased addition of the solution relative to the weight of shredded tobacco is effective, but also use of diacetin is effective and diacetin is suitable for an aerosol generating liquid.

[Table 2]

[0076]

Table 2 Prepared samples and evaluation results

Classification	Aerosolgenerating liquid	Addition of the solution relative to the weight of shredded tobacco [% by weipht]	Inhibition of flavor tasting [-]
Comparative Example 8	None	0	6.8
Comparative Example 9	Diacetin	5	4.3
Comparative Example 10		10	4.3
Comparative Example 11		20	3.5
Comparative Example 12		30	3.3
Comparative Example 13		40	4.0
Example 3		50	2.0
Example 4		70	2.0
Example 2		100	1.9
Comparative Example 14	Propylene glycol	100	6.3

<Reference Examples 1 to 8: Examination of mechanism of generation of diacetin and monoacetin hydrolysate (acetic acid) >

[0077] In order to examine the mechanism of generation of acetic acid, which is a hydrolysate of diacetin and monoacetin, to 100 mg of shredded tobacco (shredded flue-cured tobacco produced in Japan) or calcium carbonate (mock without shredded tobacco), 100 mg of diacetin, as a typical example, was added to prepare samples indicated in Table 3. The samples were evaluated for inhibition of flavor tasting and the acetic acid amount. In order to examine an effect of hydrolysis by the water amount, water was further added as indicated in Table 3. The sensory evaluation was carried out by two evaluators and "sourness" was evaluated on a 7-grade scale of 1 to 7. In the results of the present Examples, when the evaluated score on sourness was 1.5 or more, evaluators could sense the sourness, and thus the region of 1.5 or less was regarded to have an excellent suppression effect of diacetin decomposition. The acetic acid was assayed by storing the samples prepared under the conditions indicated in Table 3 for 1 week under the conditions of 22°C and humidity of 60%, placing the same in screw vials (No. 5, produced by Maruemu Corporation) and subjecting the same to extraction by shaking in a methanol solvent for 40 minutes followed by GC-MS.

[Table 3]

[0078]

Table 3 Prepared samples and evaluation/assay results

Classification	Type of shredded tobacco	Liquid	Amount of water added [mg]	Acetic acid generation [mg/pod]	Sourness [-]
Reference Example 1	Shredded flue-cured tobacco produced in Japan	Diacetin	0	1.58	3.5
Reference Example 2			5	2.74	5.75
Reference Example 3			10	3.73	7
Reference Example 4			30	6.97	7
Reference Example 5	Calcium carbonate		0	0.17	1
Reference Example 6			5	0.13	1
Reference Example 7			10	0.11	1
Reference Example 8			30	0.14	1

[0079] Fig. 3 indicates a graph illustrating the relationship between the added amount of water and the generated amount of acetic acid. From Fig. 3, the acetic acid amount in sample pods was extremely low for the samples obtained by adding diacetin to calcium carbonate regardless of the added amount of water. Meanwhile, it was found that when diacetin is added to shredded tobacco, a significant amount of acetic acid was generated in proportion to the added amount of water. From this result, it was found that when only diacetin (containing calcium carbonate) was stored, the added amount of water *per se* hardly affected decomposition of diacetin, while when diacetin was stored in the coexistence of shredded tobacco, decomposition of diacetin was promoted as the added amount of water increased and acetic acid, which is a hydrolysate of diacetin, drastically increased. Accordingly, it is speculated that although diacetin alone is hardly hydrolysed, hydrolase in shredded tobacco promotes hydrolysis of diacetin to generate a large amount of acetic acid, which is a hydrolysate of diacetin.

[0080] Finally, Fig. 4 indicates a graph illustrating the relationship between the acetic acid amount and sourness . From Fig. 4, it is found that the acetic acid amount is highly correlated with sourness and the "sourness" sensed during smoking of samples after storage is caused by an increased amount of acetic acid generated by hydrolysis of diacetin.

<Examples 5 and 6 and Comparative Examples 15 to 17: Verification of suppression method of hydrolysis>

[0081] From previous Examples, it was found that hydrolase in shredded tobacco worked as a catalyst to generate acetic acid, which is a hydrolysate of diacetin. In the present Examples, an effect of suppression of hydrolysis of diacetin was verified by, as a typical example, heat treatment which is a common manner for inactivating hydrolases.

[0082] Shredded tobacco was heated as follows: 2 g of shredded tobacco (shredded flue-cured tobacco produced in Japan) was placed in a screw vial (No. 5, produced by Maruemu Corporation) which was then covered and heated at 100°C, 120°C, 140°C or 160°C for 60 min in a hot-air circulating oven (KLO-60M, produced by Koyo Thermo Systems Co., Ltd.) . The samples were prepared in the same manner as in previous Examples, stored for 2 months under the conditions of 22°C and humidity of 60%, and the acetic acid amount was determined and the sensory evaluation was carried out. The sensory evaluation was carried out by two evaluators and "sourness" was evaluated on a 7-grade scale of 1 to 7. In the results of the present Examples, when the evaluated score on sourness was 1.5 or more, evaluators could sense the sourness, and thus the region of 1.5 or less was regarded to have an excellent suppression effect of diacetin decomposition. The acetic acid was determined by storing the samples prepared under the conditions indicated in Table 4 for 2 months under the conditions of 22°C and humidity of 60%, placing the same in screw vials (No. 5, produced by Maruemu Corporation) and subjecting the same to extraction by shaking in a methanol solvent for 40 minutes followed by GC-MS.

[Table 4]

[0083]

Table 4 Sample information and evaluation results

Classification	Type of shredded tobacco	Heating temperature [°C]	Acetic acid amount [mg/pod]	Sourness [-]	Acetic acid generation rate constant [s^{-1}]
Comparative Example 15	Shredded flue-cured tobacco produced in Japan	No heating	8.90	7	1.21×10^{-6}
Comparative Example 16		100	2.87	3.5	3.90×10^{-8}
Comparative Example 17		120	1.24	1.75	1.45×10^{-8}
Example 5		140	0.79	1	1.17×10^{-8}
Example 6		160	0.54	1	9.22×10^{-9}

[0084] Fig. 5 indicates a graph illustrating the relationship between the days of storage and the acetic acid amount in pod. From Fig. 5, it is found that when shredded tobacco is not heated, the acetic acid amount in pod significantly increases with the days of storage, while when only shredded tobacco is preliminarily subjected to heat treatment, the acetic acid amount produced in pod decreases.

[0085] When shredded tobacco is not heated, the generated amount of acetic acid drastically increases until the day 21 (3 weeks) of storage, while from day 21 to day 60 (2 months) of storage, an increment of the acetic acid amount is small, the generation rate of acetic acid by decomposition of diacetin decreases and the acetic acid amount asymptotically approaches the maximum generated-amount of acetic acid by hydrolysis of diacetin. Accordingly, the generated amount of acetic acid (8.90 mg/pod) after 2-month storage without heating of shredded tobacco can be approximated to the maximum amount (C_{max}) of acetic acid generated by hydrolysis of added-diacetin to pod, and thus a value obtained by dividing the acetic acid amount (C) in pod by the maximum amount (C_{max}) of acetic acid generated is defined to be the proportion of acetic acid generated (C/C_{max}).

[0086] From Fig. 6 which indicates the relationship of the proportion of generated acetic acid relative to days of storage, generation of acetic acid (decomposition of diacetin) until day 21 of storage behaves according to first-order reaction. Therefore, the acetic acid generation rate constant k organized relative to the days t of storage can be represented by the following first-order reaction rate equation.

$$kt = -\ln\left(1 - \frac{C}{C_{max}}\right)$$

[0087] Fig. 7 indicates a graph obtained by plotting the above equation for each heating temperature condition. From Fig. 7, the acetic acid generation rate constant k of shredded tobacco was calculated from the slope of the plot from day 1 to day 21 of storage. Because of a small amount of acetic acid contained in shredded tobacco, the graph shown in Fig. 7 does not start from the origin.

[0088] From Table 4 in which the acetic acid generation rate constant k of shredded tobacco was calculated, it was found that when shredded tobacco has an acetic acid generation rate constant of $1.17 \times 10^{-8}\ s^{-1}$ or less, the acetic acid amount was extremely low even after 2-month storage of samples and sourness could not be noticed from the sensory evaluation results, and thus hydrolysis of diacetin can be suppressed by using shredded tobacco having an acetic acid generation rate constant of $1.17 \times 10^{-8}\ s^{-1}$ or less with diacetin. It can be approximately calculated that the acetic acid generation rate constant corresponding to the sourness of 1.5 is $1.25 \times 10^{-8}\ s^{-1}$, and thus it is speculated that a suppression effect of decomposition is sufficiently secured when the tobacco material has an acetic acid generation rate constant of $1.25 \times 10^{-8}\ s^{-1}$ or less.

[Industrial Applicability]

[0089] The tobacco filler of the present invention can be used for smoking by filling a container such as a pod for a non-combustion type heat smoking article.

Claims

1. A tobacco filler for a non-combustion type heat smoking article comprising a tobacco material and an aerosol generating liquid, wherein:

   the aerosol generating liquid contains at least one of diacetin and monoacetin;
   the tobacco material has an acetic acid generation rate constant of 1.25×10^{-8} s^{-1} or less and an added amount of the aerosol generating liquid is 50% by weight to 300% by weight relative to the weight of the tobacco material.

2. The tobacco filler according to claim 1, wherein the aerosol generating liquid contains both diacetin and monoacetin.

3. The tobacco filler according to claim 1 or 2, wherein the aerosol generating liquid contains 50% by weight or more in total of diacetin and monoacetin relative to the aerosol generating liquid.

4. A pod for a non-combustion type heat smoking article, comprising the tobacco filler according to any one of claims 1 to 3 filling therein.

5. A non-combustion type heat smoking article, comprising the tobacco filler according to any one of claims 1 to 3.

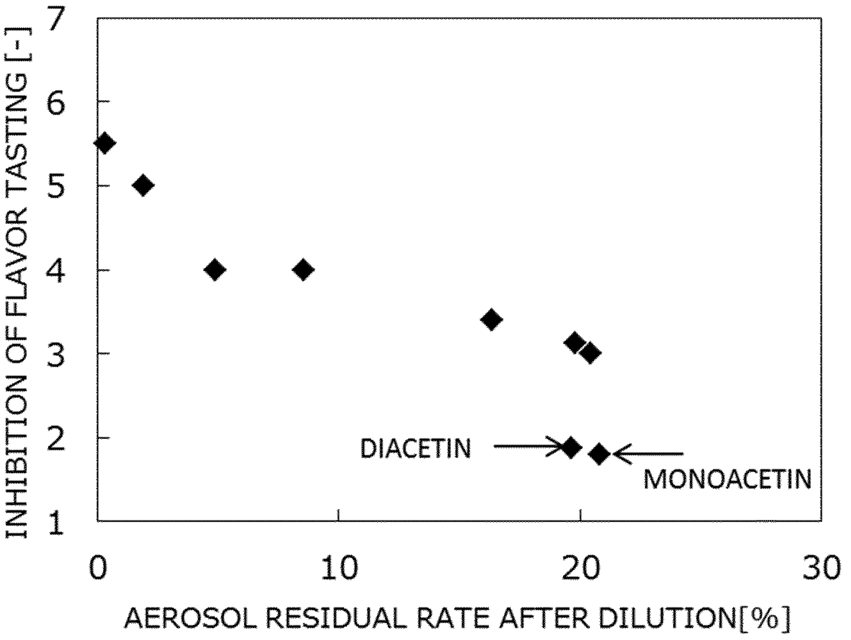
INHIBITION OF FLAVOR TASTING [-]
1
2
3
4
5
6
7
DIACETIN
MONOACETIN
0
10
20
30
AEROSOL RESIDUAL RATE AFTER DILUTION[%]

Fig.1

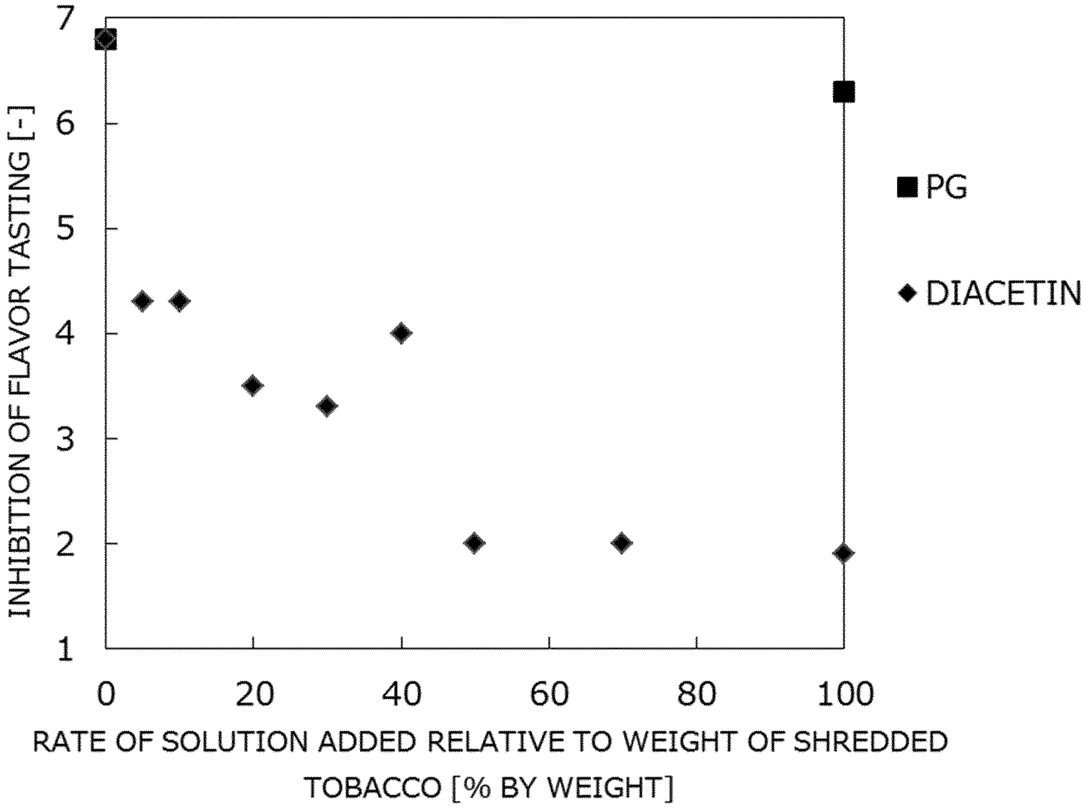
INHIBITION OF FLAVOR TASTING [-]
1
2
3
4
5
6
7
■ PG
◆ DIACETIN
0
20
40
60
80
100
RATE OF SOLUTION ADDED RELATIVE TO WEIGHT OF SHREDDED TOBACCO [% BY WEIGHT]

Fig.2

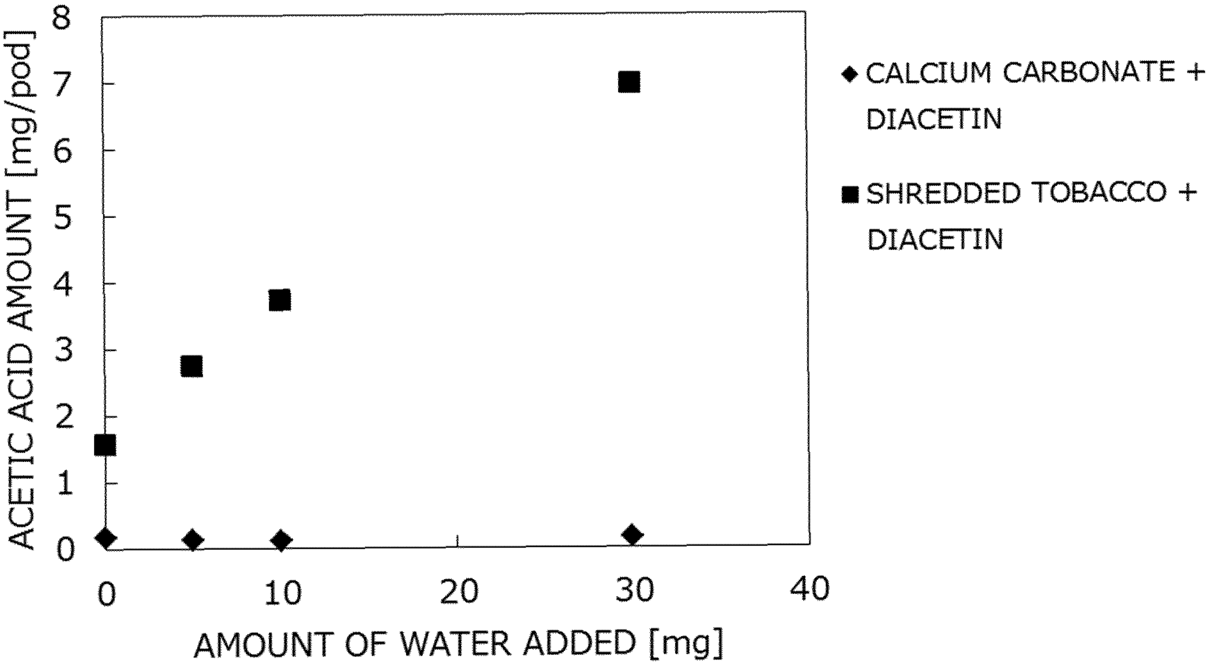
ACETIC ACID AMOUNT [mg/pod]
8
7
6
5
4
3
2
1
0
0
10
20
30
40
AMOUNT OF WATER ADDED [mg]
◆ CALCIUM CARBONATE + DIACETIN
■ SHREDDED TOBACCO + DIACETIN

Fig.3

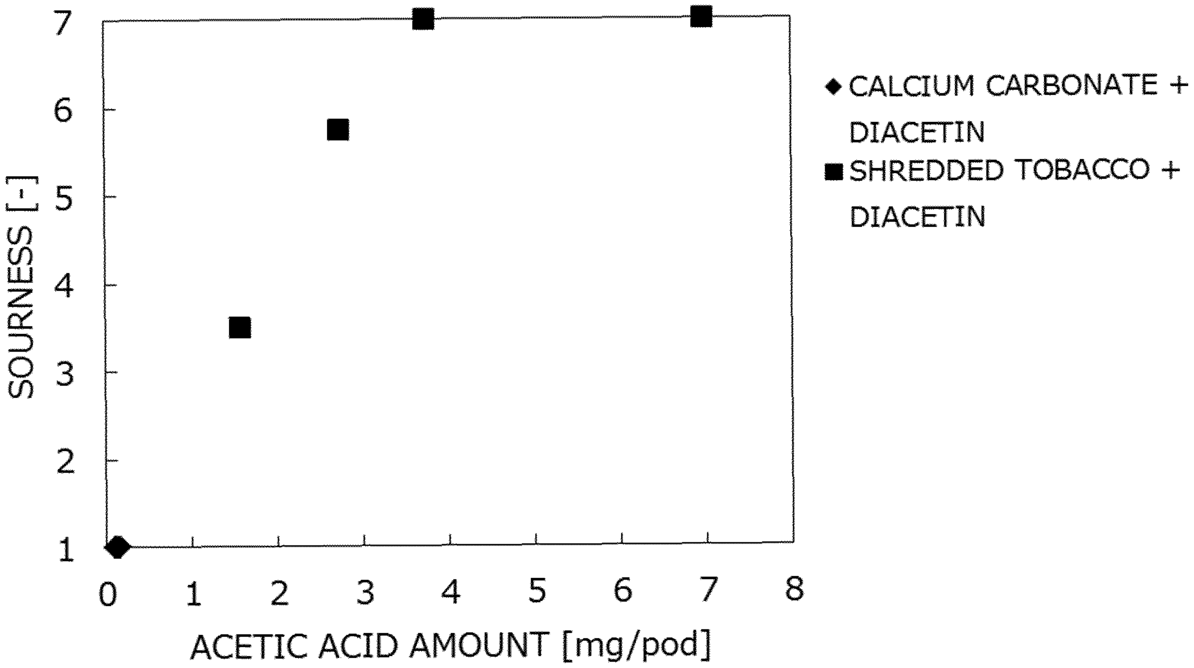
SOURNESS [-]
7
6
5
4
3
2
1
0
1
2
3
4
5
6
7
8
ACETIC ACID AMOUNT [mg/pod]
◆ CALCIUM CARBONATE + DIACETIN
■ SHREDDED TOBACCO + DIACETIN

Fig.4

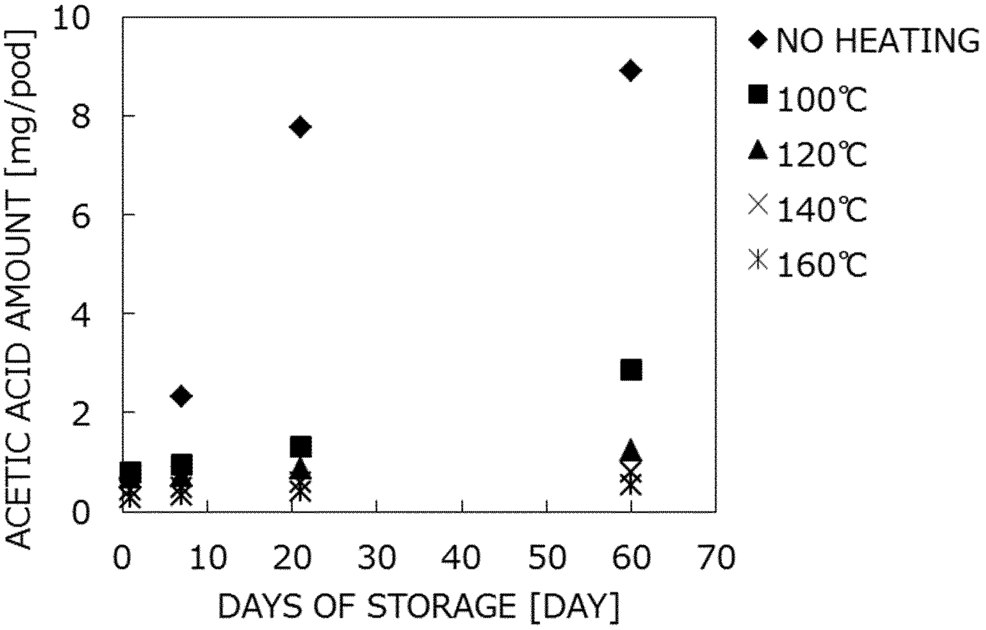
ACETIC ACID AMOUNT [mg/pod]
10
8
6
4
2
0
0
10
20
30
40
50
60
70
DAYS OF STORAGE [DAY]
◆ NO HEATING
■ 100℃
▲ 120℃
× 140℃
✳ 160℃

Fig.5

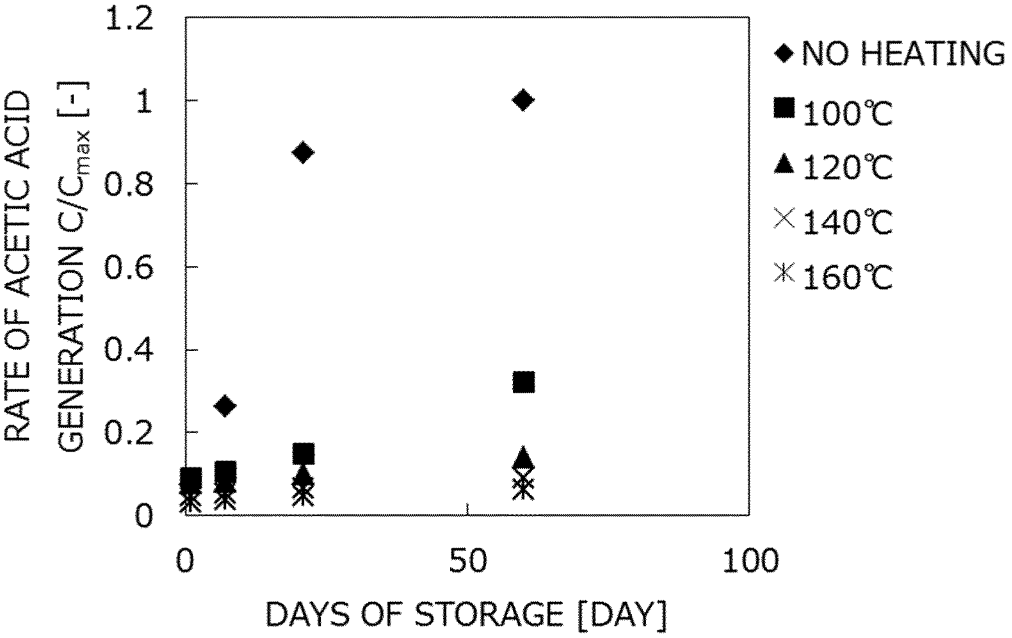
RATE OF ACETIC ACID GENERATION C/C_{max} [-]
1.2
1
0.8
0.6
0.4
0.2
0
0
50
100
DAYS OF STORAGE [DAY]
◆ NO HEATING
■ 100℃
▲ 120℃
× 140℃
✳ 160℃

Fig.6

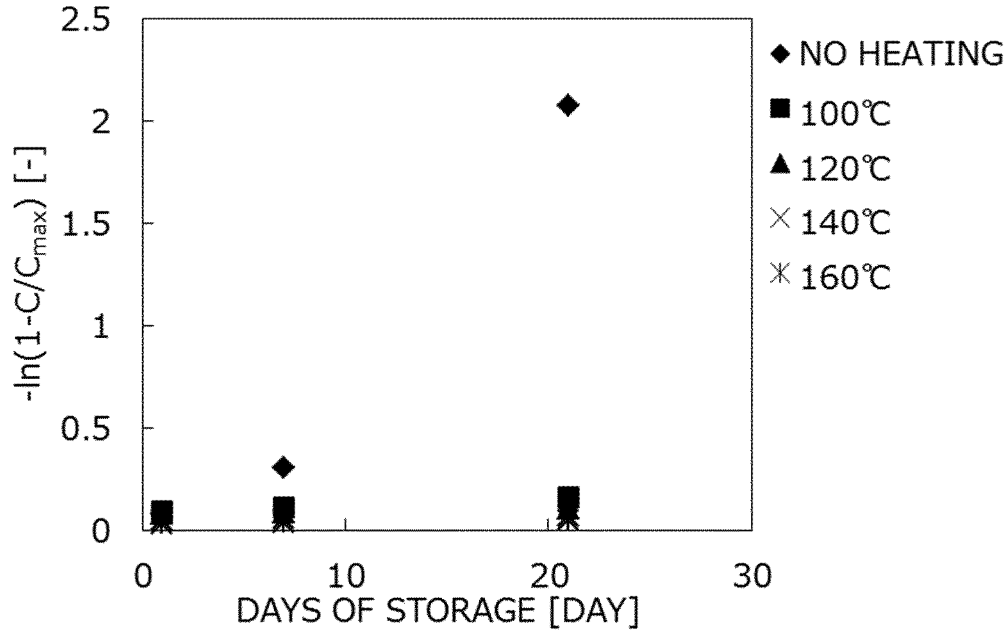
2.5
2
1.5
1
0.5
0
-ln(1-C/C_max) [-]
0
10
20
30
DAYS OF STORAGE [DAY]
◆ NO HEATING
■ 100℃
▲ 120℃
× 140℃
✳ 160℃

Fig.7

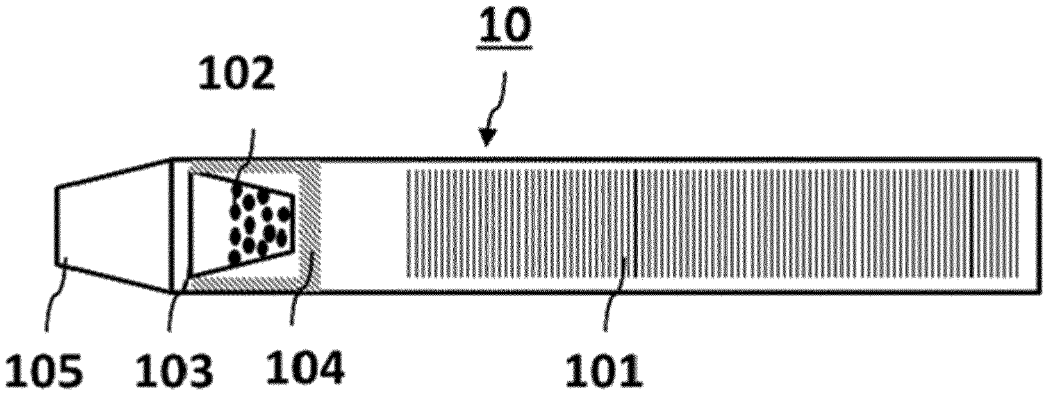
10
102
105
103
104
101

Fig.8

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2016/071526

A. CLASSIFICATION OF SUBJECT MATTER
A24F47/00(2006.01)i

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
A24F47/00

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

Jitsuyo Shinan Koho	1922-1996	Jitsuyo Shinan Toroku Koho	1996-2016
Kokai Jitsuyo Shinan Koho	1971-2016	Toroku Jitsuyo Shinan Koho	1994-2016

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2013-507906 A (British American Tobacco (Investments) Ltd.), 07 March 2013 (07.03.2013), paragraphs [0015] to [0016] & US 2012/0298123 A1 paragraphs [0016] to [0017] & GB 918129 D & WO 2011/045609 A1 & EP 2488054 A & CN 102548431 A & KR 10-2012-0101654 A	1-5
A	JP 2007-70534 A (Daicel Chemical Industries, Ltd.), 22 March 2007 (22.03.2007), paragraph [0013] (Family: none)	1-5

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

* Special categories of cited documents:

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

Date of the actual completion of the international search
16 September 2016 (16.09.16)

Date of mailing of the international search report
27 September 2016 (27.09.16)

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)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2016/071526

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2012-504692 A (E.I. Du Pont de Nemours & Co.), 23 February 2012 (23.02.2012), paragraph [0317] & US 2010/0086510 A1 paragraph [0382] & WO 2010/039956 A1 & EP 2342324 A & CN 102239255 A	1-5

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

REFERENCES CITED IN THE DESCRIPTION

Patent documents cited in the description

- JP S63148975 B **[0004]**