



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

(43) Date of publication:
08.07.2015 Bulletin 2015/28

(51) Int Cl.:
A24B 15/30 ^(2006.01) **A24B 3/12** ^(2006.01)
A24D 1/02 ^(2006.01) **C11B 9/00** ^(2006.01)

(21) Application number: **13833905.6**

(86) International application number:
PCT/JP2013/072774

(22) Date of filing: **26.08.2013**

(87) International publication number:
WO 2014/034619 (06.03.2014 Gazette 2014/10)

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

(72) Inventors:
• **TANAKA, Yasuo**
Tokyo 130-8603 (JP)
• **OTOKAWA, Takuya**
Tokyo 130-8603 (JP)

(30) Priority: **31.08.2012 JP 2012192248**

(74) Representative: **Isarpatent**
Patentanwälte Behnisch Barth Charles
Hassa Peckmann & Partner mbB
Friedrichstrasse 31
80801 München (DE)

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

(54) **FRAGRANCE-CARRYING CIGARETTE CONSTITUENT MEMBER AND CIGARETTE CONTAINING SAME**

(57) A flavor-carrying cigarette constituent member, which is obtained by: applying a flavor composition in a sol state to a surface of a cigarette constituent member, the flavor composition containing a flavor, water, and a polysaccharide selected from the group consisting of carrageenan, gellan gum, and a combination of gellan gum

and tamarind gum; then cooling the flavor composition-applied cigarette constituent member to a temperature of 0 to 40°C to cause gelation of the flavor composition; and then heat-drying the flavor composition-applied cigarette constituent member.

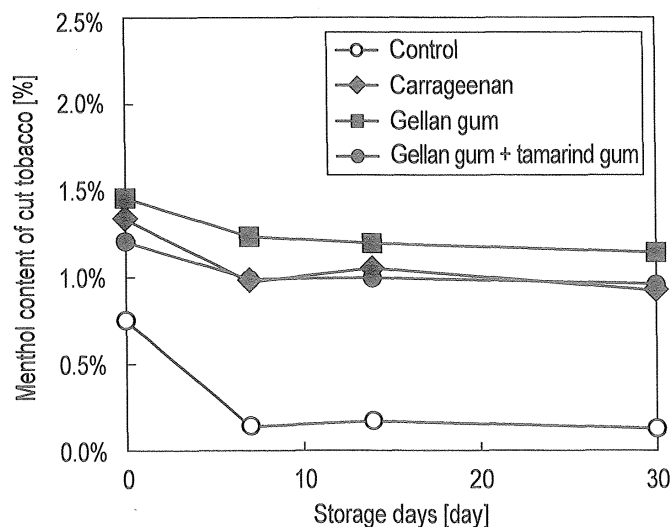


FIG. 1

Description

Technical Field

5 **[0001]** The present invention relates to a flavor-carrying cigarette constituent member, and a cigarette comprising the same.

Background Art

10 **[0002]** If a volatile flavor component such as menthol is added to cut tobacco in a solution state, the flavor component is dissipated during long-term storage and the flavor effect does not last. In order to solve such a problem, various reports have been made.

[0003] Patent Documents 1 to 3 report an example in which the flavor component is placed in cut tobacco, which is a burning site, or a cigarette paper, which wraps the cut tobacco.

15 **[0004]** Patent Document 1 discloses that a cigarette paper wrapping a tobacco filler is coated with a flavor material in which a flavor component is incorporated into a three-dimensional network of glucan molecules. The cigarette of Patent Document 1 has a good flavor-retaining property, because the flavor component is fixed and retained with incorporated into the three-dimensional network of the glucan molecules. The flavor component, however, exists in a relatively small amount (20% by weight or less) in the glucan molecules. Accordingly, in a case of a flavor component such as menthol, 20 which requires a relatively large addition amount, the blending amount of the flavor material to the cigarette becomes larger.

[0005] Patent Document 2 discloses that a "stabilized flavor substance which is stable up to 180°C" is prepared by mixing a liquid flavor with a carrageenan sol; dropping the mixture into an ionic solution (a solution containing potassium ions) to prepare particulate gel; and drying the gel in air. According to the method of Patent Document 2, however, a 25 long time and large facilities are required in order to prepare a large amount of the material, because the particulate gel is dried in air.

[0006] Patent Document 3 reports that a sheet containing a flavor component with coated with a gel of polysaccharide is produced by drying a slurry containing the flavor component such as menthol and the polysaccharide; and the sheet is cut and the cut pieces are added to cut tobacco. The flavor-containing sheet of Patent Document 3 has a high flavor 30 content because the flavor component is coated with the gel of polysaccharide and thus it is difficult to volatilize the flavor component.

Prior Art Document

35 Patent Document

[0007]

40 Patent Document 1: Jpn. Pat. Appln. KOKAI Publication No. 9-28366
Patent Document 2: Jpn. PCT National Publication No. 11-509566
Patent Document 3: WO 2009/142159

Summary of Invention

45 Technical Problem

[0008] The present inventors found that, when cut pieces of the flavor-containing sheet having a high flavor content are incorporated into a cigarette as a tobacco filler in a small amount (for example, a blending ratio of 2% by weight) in accordance with Patent Document 3, a non-uniform flavor distribution sometimes occurs in a cigarette rod. In order to 50 improve the defect, the present invention aims at providing a flavor-carrying cigarette constituent member which has a high flavor stability and which can uniformly blend the flavor in a cigarette when it is incorporated into the cigarette.

Solution to Problem

55 **[0009]** The present inventors found that, when a slurry containing a specific polysaccharide and a flavor is applied to a cigarette constituent member such as a tobacco filler or cigarette paper, a high flavor stability can be obtained, the flavor can be uniformly blended in the cigarette, and variation in the blending amount of the flavor between cigarettes can be suppressed. As a result, the present inventors completed the present invention.

[0010] According to a first aspect of the present invention, a flavor-carrying cigarette constituent member is provided which is obtained by: applying a flavor composition in a sol state to a surface of a cigarette constituent member, the flavor composition containing a flavor, water, and a polysaccharide selected from the group consisting of carrageenan, gellan gum, and a combination of gellan gum and tamarind gum; then cooling the flavor composition-applied cigarette constituent member to a temperature of 0 to 40°C to cause gelation of the flavor composition; and then heat-drying the flavor composition-applied cigarette constituent member.

[0011] According to a preferable embodiment, the heat-drying is performed so that the surface temperature of the flavor composition is kept at 70 to 100°C.

[0012] According to a specific embodiment, the cigarette constituent member is a tobacco filler or a cigarette paper. According to a more specific embodiment, the tobacco filler is cut tobacco or sheet tobacco.

[0013] According to a second aspect of the present invention, a cigarette comprising the flavor-carrying cigarette constituent member of the present invention is provided.

Effects of Invention

[0014] The flavor-carrying cigarette constituent member of the present invention has a high flavor-retaining property after storage, and an advantage that the flavor can be uniformly blend in the cigarette (more accurately, in a site into which the flavor-carrying cigarette constituent member is incorporated), when it is incorporated into the cigarette. In other words, when a cigarette is produced using the flavor-carrying cigarette constituent member of the present invention, a non-uniform flavor distribution can be suppressed in a cigarette rod. As a result, the cigarette produced using the flavor-carrying cigarette constituent member of the present invention can continuously and stably deliver the flavor over the full smoking time, whereby the same smoking flavor and taste can be provided to smokers over the full smoking time.

[0015] In addition, the flavor-carrying cigarette constituent member of the present invention also has an advantage that variation in the flavor content can be suppressed between cigarettes when multiple cigarettes are produced using the member, because the flavor is uniformly blended in the cigarette.

Brief Description of Drawings

[0016]

FIG. 1 is a graph showing a post-storage flavor-retaining property of menthol in cut tobacco whose surface is applied with a menthol-containing composition.

FIG. 2 is a graph showing a post-storage flavor-retaining property of menthol in a cigarette comprising the cut tobacco whose surface is applied with a menthol-containing composition.

FIG. 3 is a graph showing a post-storage flavor-retaining property of menthol in sheet tobacco whose surface is applied with a menthol-containing composition.

FIG. 4 is a graph showing a post-storage flavor-retaining property of menthol in a cigarette comprising the sheet tobacco whose surface is applied with a menthol-containing composition.

FIG. 5 is a graph showing a post-storage flavor-retaining property of menthol in a cigarette paper whose surface is applied with a menthol-containing composition.

FIG. 6 is a graph showing a post-storage flavor-retaining property of menthol in a cigarette comprising the cigarette paper whose surface is applied with a menthol-containing composition.

FIG. 7A is a graph showing a change in a viscosity followed by a fall in temperature of a carrageenan-containing liquid.

FIG. 7B is a graph showing a change in a viscosity followed by a rise in temperature of the carrageenan-containing liquid.

FIG. 8A is a graph showing a change in a viscosity followed by a fall in temperature of a gellan gum-containing liquid.

FIG. 8B is a graph showing a change in a viscosity followed by a rise in temperature of the gellan gum-containing liquid.

FIG. 9A is a graph showing a change in a viscosity followed by a fall in temperature of a liquid containing gellan gum and tamarind gum.

FIG. 9B is a graph showing a change in a viscosity followed by a rise in temperature of the liquid containing gellan gum and tamarind gum.

Description of Embodiments

[0017] The present invention will be explained below. The following explanations are intended to describe the present invention in detail, and are not intended to limit the present invention.

[0018] The flavor-carrying cigarette constituent member of the present invention is obtained by:

applying a flavor composition in a sol state to a surface of a cigarette constituent member, the flavor composition containing a flavor, water, and a polysaccharide selected from the group consisting of carrageenan, gellan gum, and a combination of gellan gum and tamarind gum; then
 5 cooling the flavor composition-applied cigarette constituent member to a temperature of 0 to 40°C to cause gelation of the flavor composition; and then
 heat-drying the flavor composition-applied cigarette constituent member.

[0019] A flavor used in the present invention is not limited, so long as it is a flavor used in smoking articles. Any type of flavor can be used. Examples of the flavor mainly include menthol; leaf tobacco extract; natural plant flavors (for
 10 example, cinnamon, sage, herb, chamomile, kudzu (*Pueraria lobata*), sweet hydrangea leaf, clove, lavender, cardamom, caryophyllus, nutmeg, bergamot, geranium, honey essence, rose oil, lemon, orange, cassia bark, caraway, jasmine, ginger, coriander, vanilla extract, spearmint, peppermint, cassia, coffee, celery, cascarilla, sandalwood, cocoa, ylang-ylang, fennel, anise, licorice, St. John's bread, prune extract, and peach extract); saccharides (for example, glucose, fructose, isomerized saccharide, and caramel); cocoas (for example, powder and extract); esters (for example, isoamyl
 15 acetate, linalyl acetate, isoamyl propionate, and linalyl butyrate); ketones (for example, menthon, ionone, damascenone, and ethyl maltol); alcohols (for example, geraniol, linalool, anethole, and eugenol); aldehydes (for example, vanillin, benzaldehyde, and anisaldehyde); lactones (for example, γ -undecalactone and γ -nonalactone); animal flavors (for example, musk, ambergris, civet, and castoreum); and hydrocarbons (for example, limonene and pinene). A flavor which is easily dispersed in a solvent by addition of an emulsifier, such as a hydrophobic flavor and oil-soluble flavor, can be
 20 preferably used. Such a flavor may be used alone or in combination.

[0020] Hereinafter, the present invention will be explained with an example where menthol is used as a flavor.

1. Menthol-Containing Composition

[0021] In one embodiment, a flavor-carrying cigarette constituent member of the present invention is obtained by
 25 applying a flavor composition containing menthol and a polysaccharide selected from the group consisting of carrageenan, gellan gum, and a combination of gellan gum and tamarind gum (hereinafter referred to as a "menthol-containing composition") to a cigarette constituent member. The flavor-carrying cigarette constituent member of the present invention is preferably obtained by applying the flavor composition to a surface of the cigarette constituent member.

[0022] The menthol-containing composition used in the present invention is in the state of a slurry having fluidity at a
 30 heating temperature (for example, 60 to 90°C) before it is dried.

[0023] The composition of the menthol-containing composition (slurry) can be as follows: for example, 200 to 500 grams of the polysaccharide, 1000 to 2500 grams of menthol, and 80 to 200 ml of a 2 to 10% by weight emulsifier solution, per 10 liters of water. The menthol-containing composition (slurry) has typically a water content of 70 to 95%
 35 by weight, preferably 80 to 90% by weight. In the menthol-containing composition, a ratio (weight ratio) of the polysaccharide to menthol can be from 1:1 to 1:10.

[0024] In the present invention, the polysaccharide has a property such that it is fixing micelles of a flavor such as menthol to coat the flavor with it, by causing gelation of the aqueous polysaccharide solution by cooling. As the polysaccharide having the property, a polysaccharide selected from the group consisting of carrageenan, gellan gum, and a
 40 combination of gellan gum and tamarind gum can be used. In the present invention, when carrageenan is used as the polysaccharide, another polysaccharide such as gellan gum or tamarind gum may be included in the menthol-containing composition in an amount smaller than that of carrageenan. Similarly, when gellan gum is used as the polysaccharide, another polysaccharide such as carrageenan may be included in the menthol-containing composition in an amount smaller than that of gellan gum.

[0025] In the present invention, 1-menthol can be used as menthol.

[0026] In the present invention, a naturally derived emulsifier such as lecithin, specifically Sun Lecithin A-1 (Taiyo Kagaku Co., Ltd.), can be used as the emulsifier.

[0027] In the present invention, the menthol-containing composition (slurry) can be prepared by a method comprising:
 50 a step (i) of mixing the polysaccharide with water and heating the mixture to prepare an aqueous solution of the polysaccharide; and a step (ii) of adding menthol and the emulsifier to the aqueous solution, and kneading and emulsifying the mixture.

[0028] The step (i) can be specifically performed by adding the polysaccharide to water little by little while being stirred, thereby dissolving the polysaccharide. The heating temperature in this step can be from 60 to 90°C, preferably from 75 to 85°C. The step (ii) can be performed by a known emulsification technique using a homogenizer, because the menthol-containing composition (slurry) has a viscosity of about 10,000 mPas or less (a sol state) at the above heating temperature,
 55 by which the emulsification is not inhibited.

2. Application of Menthol-Containing Composition to Cigarette Constituent Member

[0029] In the present invention, the menthol-containing composition is applied to the cigarette constituent member, preferably a surface of the cigarette constituent member.

[0030] Specifically, in the present invention, the cigarette constituent member is a constituent member disposed at a position which does not involve contact with a mouth or an oral cavity of a smoker when it is incorporated into the cigarette. In other words, the cigarette constituent member is a constituent member disposed at a position through which the flavor such as menthol can be delivered together with mainstream smoke by combustion at the time of smoking, when it is incorporated into the cigarette.

[0031] More specifically, the cigarette constituent member is a tobacco filler or a cigarette paper. Still more specifically, the tobacco filler is cut tobacco or sheet tobacco.

[0032] The method of applying the menthol-containing composition to the cigarette constituent member is not particularly limited so long as the menthol-containing composition (slurry) can be uniformly applied to the cigarette constituent member. The menthol-containing composition (slurry) may be added to or coated on the surface of the cigarette constituent member, the menthol-containing composition (slurry) may be sprayed on the surface of the cigarette constituent member, or the cigarette constituent member may be immersed in the menthol-containing composition (slurry).

[0033] When the menthol-containing composition slurry (in the state of a sol at about 60 to 90°C) is applied to cut tobacco, the menthol-containing composition (slurry) can be added directly to the surface of the cut tobacco using a transfer pump, or the composition can be sprayed on the cut tobacco using a nozzle sprayer. Alternatively, the cut tobacco may be immersed in the menthol-containing composition slurry to apply the menthol-containing composition (slurry) to the cut tobacco.

[0034] When the menthol-containing composition slurry (in the state of a sol at about 60 to 90°C) is applied to sheet tobacco, the menthol-containing composition (slurry) can be added by extrusion to the surface of the sheet tobacco using a slit feeder, or the composition can be coated on the surface of the sheet tobacco using a film applicator. Alternatively, spraying or immersing can be performed according to the same manner as used in the application to the cut tobacco.

[0035] When the menthol-containing composition slurry (in the state of a sol at about 60 to 90°C) is applied to cigarette paper, the menthol-containing composition (slurry) can be added by extrusion using a slit feeder or coated using a film applicator as described above. Alternatively, spraying or immersing can be performed according to the same manner as used in the application to the cut tobacco.

[0036] In a case of the cut tobacco, the menthol-containing composition slurry can be applied in an amount of 5 to 40 parts by weight, based on 100 parts by weight of the cut tobacco, preferably 15 to 25 parts by weight. In a case of the sheet tobacco, the menthol-containing composition slurry can be applied in an amount of 50 to 400 parts by weight, based on 100 parts by weight of the sheet tobacco, preferably 100 to 200 parts by weight. In a case of the cigarette paper, the menthol-containing composition slurry can be applied in an amount of 10 to 70 parts by weight, based on 100 parts by weight of the cigarette paper, preferably 30 to 50 parts by weight.

[0037] Before drying the cigarette constituent member to which the menthol-containing composition slurry has been applied, it is preferable to once cool the cigarette constituent member so that the slurry becomes a temperature enabling sufficient gelation of the slurry (40°C or lower) and avoiding breaking of emulsion due to freezing (0°C or higher). Specifically, it is preferable to once cool the cigarette constituent member so that the slurry becomes a temperature of 0 to 40°C, preferably 0 to 30°C, and more preferably 15 to 25°C. Here, the menthol-containing composition slurry, before it is cooled, has a temperature of 60 to 90°C, preferably 75 to 85°C, and is in the state of a sol. Such preliminary cooling can be performed by blowing air or applying cold air (for example, 10°C) generated in a spot cooler (for example, Suiden SS-25DD-1) to the cigarette constituent member (to which the menthol-containing composition slurry has been applied) for 2 to 3 minutes. Alternatively, the preliminary cooling may be performed by bringing the cigarette constituent member (to which the menthol-containing composition slurry has been applied) into contact with a tube through which a cooling medium (for example, 10°C) generated in a cold or warm water generator (a chiller, for example, PCU-1600R, Apiste Corporation) is passed, for 1 to 2 minutes. Alternatively, the preliminary cooling may be performed by allowing the cigarette constituent member (to which the menthol-containing composition slurry has been applied) to stand at room temperature.

[0038] Once the aqueous solution of polysaccharide used in the present invention is cooled to cause gelation, the obtained gel has a property of being capable of maintaining the gel state without easily returning to a sol state even if the temperature is raised afterward to reach the gel transition temperature (see Examples described below). Utilizing the property described above, the menthol-containing composition slurry is preliminarily cooled before it is dried. As a result, the polysaccharide contained in the menthol-containing composition (which has been preliminarily cooled but has not been dried yet) is hard to solate even if the temperature is raised at the time of drying, and the menthol coated with the polysaccharide is particularly difficult to volatilize. Therefore, in the present invention, owing to the preliminary cooling, it is possible to expose the menthol-containing composition to heat in the subsequent drying step, whereby a short time

drying can be realized.

[0039] The cigarette constituent member (to which the menthol-containing composition has been applied) can be heat-dried by any type of heat-drying method such as hot air drying or infrared heat drying.

[0040] The menthol-containing composition which has been applied to the cigarette constituent member can be dried so that a sample temperature is kept at 100°C or lower, preferably 70 to 100°C in the entire drying step. In order to keep the sample temperature at 100°C or lower, a drying temperature of 100°C to 130°C is preferably employed. In order to keep the sample temperature at 70 to 100°C, a drying temperature of 100°C to 130°C is preferably employed. Here, the "sample temperature" refers to a surface temperature of the menthol-containing composition in the drying step, and the "drying temperature" refers to a temperature in the dryer. The temperature in the dryer is equal to the hot air temperature, in a case of the hot air drying.

[0041] Preferably, the drying includes drying at a drying temperature of 100°C or higher (preferably from 100°C to 130°C) for one fourth or more of the total drying time. The drying can be performed for 20 minutes or less of the total drying time until the water content of the flavor-carrying cigarette constituent member reaches less than 10%. Preferably, the drying at a drying temperature of 100°C or higher is performed in the initial drying stage, and after that the drying at a drying temperature equal to or less than the initial drying temperature (preferably 70°C or higher and less than 100°C) is performed.

[0042] The drying temperature is set so that the sample temperature is kept at 100°C or lower at all times in the entire drying step, and the drying temperature may be constant over the entire drying step or may be varied during the drying step. When the drying temperature is varied, the drying is preferably performed by an initial drying at a high drying temperature of 100°C or higher and a subsequent latter drying at a low drying temperature of lower than 100°C. In the present specification, the "initial drying" refers to a drying using a high temperature of 100°C or higher in the first stage of the drying step, and the "latter drying" refers to a drying subsequent to the initial drying, using a low temperature of lower than 100°C. When the initial drying at a high temperature is performed in combination with the latter drying at a low temperature, the sample temperature does not become too high, the sample temperature is kept at 100°C or lower at all times in the entire drying step, and the drying time can be advantageously shortened.

[0043] More preferably, the drying can be performed by the initial drying at a drying temperature of 100°C or higher for one fourth or more of the total drying time; and then the latter drying at a drying temperature of lower than 100°C for one fourth or more of the total drying time. The drying can be performed for 20 minutes or less of the total drying time until the water content of the flavor-carrying cigarette constituent member reaches a predetermined value (specifically, until the water content reaches about 12% by weight or less in the case of the flavor-carrying cigarette constituent member obtained by applying the menthol-containing composition to cut tobacco, and until the water content reaches about 10% by weight or less in the case of the flavor-carrying cigarette constituent member obtained by applying the menthol-containing composition to sheet tobacco or cigarette paper).

[0044] As described above, when the initial drying at a high temperature is performed in combination with the latter drying at a low temperature, the flavor-carrying cigarette constituent member of the present invention has a particularly high menthol content after the preparation, and can keep the menthol content at a particularly high level even after the storage.

[0045] When the hot air drying is performed, the initial drying can be performed, for example, at a hot air temperature of 100°C or higher and 130°C or lower for 4 to 6 minutes, and the latter drying can be performed, for example, at a hot air temperature of 70°C or higher and less than 100°C for 4 to 6 minutes. The air flow can be set, for example, at 3 to 20 m/second. The total drying time is 20 minutes or less, preferably from 10 to 18 minutes.

[0046] The conditions (temperature, time, and air flow) in the initial drying and the latter drying can be appropriately set, for example, within the above range.

[0047] The drying temperature in the initial drying may be constant or may be varied so that the temperature is sequentially dropped between 100°C and 130°C. The drying temperature in the latter drying may also be constant, or may be varied so that the temperature is sequentially dropped between 70°C and less than 100°C. For example, a dryer used in the Examples described below has three drying chambers, and a sample is conveyed in the order of the first, second, and third chambers by a belt conveyor. Thus, the first and second chambers may be used for the initial drying at the same or different temperatures (100°C or higher) and the third chamber may be used for the latter drying (lower than 100°C). Alternatively, the first chamber may be used for the initial drying (100°C or higher) and the second and third chambers may be used for the latter drying at the same or different temperatures (lower than 100°C).

[0048] The drying is performed preferably until the water content of the flavor-carrying cigarette constituent member reaches a predetermined value (specifically, until the water content reaches about 12% by weight or less in the case of the flavor-carrying cigarette constituent member obtained by applying the menthol-containing composition to cut tobacco, and until the water content reaches about 10% by weight or less in the case of the flavor-carrying cigarette constituent member obtained by applying the menthol-containing composition to sheet tobacco or cigarette paper). The water content herein refers to a value obtained by a measurement method described in the Examples below.

3. Cigarette

[0049] The present invention provides a cigarette comprising the flavor-carrying cigarette constituent member of the present invention. The cigarette of the present invention has the same structure as that of an ordinary cigarette except that the flavor-carrying cigarette constituent member of the present invention is used instead of a constituent member of the ordinary cigarette. In a specific embodiment, the cigarette of the present invention comprises the cut tobacco to which the flavor composition of the present invention is applied, the sheet tobacco to which the flavor composition of the present invention is applied, or the cigarette paper to which the flavor composition of the present invention is applied. The cigarette of the present invention may comprise a combination of the flavor-carrying cigarette constituent members of the present invention. For example, the cigarette may comprise the cut tobacco to which the flavor composition of the present invention is applied in combination with the sheet tobacco to which the flavor composition of the present invention is applied. Alternatively, the cigarette may comprise the cut tobacco to which the flavor composition of the present invention is applied in combination with the cigarette paper to which the flavor composition of the present invention is applied.

[0050] The cut tobacco to which the flavor composition of the present invention is applied can be incorporated into a cigarette in a content of 20 to 100% by weight of the entire tobacco filler, when the tobacco filler included in one cigarette is defined as 100% by weight. The sheet tobacco to which the flavor composition of the present invention is applied can be incorporated into a cigarette in a content of 10 to 40% by weight of the entire tobacco filler, when the tobacco filler included in one cigarette is defined as 100% by weight.

Examples

[0051] The present invention will be explained by means of the Examples below.

[1] Preparation of Menthol-Containing Compositions (Slurry) 1 to 3

[0052]

Composition of Menthol-Containing Composition (Slurry) 1 (Carrageenan was used as a polysaccharide.)

Water 10 liters

Carrageenan (CS-530/San-Ei Gen F.F.I., Inc.) 500 grams

Lecithin (Sun Lecithin A-1/Taiyo Kagaku Co., Ltd.) 200 milliliters (5% aqueous solution)

Menthol (Takasago International Corporation) 2500 grams

Composition of Menthol-Containing Composition (Slurry) 2 (Gellan gum was used as a polysaccharide.)

Water 10 liters

Gellan Gum (KELCOGEL/San-Ei Gen F.F.I., Inc.) 300 grams

Lecithin (Sun Lecithin A-1/Taiyo Kagaku Co., Ltd.) 120 milliliters (5% aqueous solution)

Menthol (Takasago International Corporation) 1500 grams

Composition of Menthol-Containing Composition (Slurry) 3 (Gellan gum and tamarind gum are used as a polysaccharide.)

Water 10 liters

Gellan Gum (KELCOGEL/San-Ei Gen F.F.I., Inc.) 150 grams

Tamarind Gum (VIS TOP D-2032/San-Ei Gen F.F.I., Inc.) 150 grams

Lecithin (Sun Lecithin A-1/Taiyo Kagaku Co., Ltd.) 120 milliliters (5% aqueous solution)

Menthol (Takasago International Corporation) 1500 grams

[0053] The menthol-containing compositions (slurry) 1 to 3 having the above composition were prepared as follows:

Water (10 L) was kept at 80°C, and the polysaccharide (carrageenan (500 g) in the case of the menthol-containing composition 1, gellan gum (300 g) in the case of the menthol-containing composition 2, or gellan gum (150 g) and tamarind gum (150 g) in the case of the menthol-containing composition 3) was dissolved in the water little by little so as not to form lumps (the time required was about 20 minutes), while the mixture was stirred using a mixer (PRIMIX T.K. AUTO MIXER Model 40/equipped with a solution stirring rotor/2000 rpm). After that, menthol (2500 g in the case of the menthol-containing composition 1, and 1500 g in the cases of the menthol-containing compositions 2 and 3) was added.

[0054] The stirring mixer was changed to a homogenizer (PRIMIX T.K. AUTO MIXER Model 40/equipped with a rotor stator head/4000 rpm) and the mixture was emulsified for 10 minutes. Then, lecithin (200 mL of 5% aqueous solution

in the case of the menthol-containing composition 1, and 120 mL of 5% aqueous solution in the cases of the menthol-containing compositions 2 and 3) was added thereto, and the emulsification was continued for 10 minutes to obtain a menthol-containing composition (slurry).

[0055] The obtained menthol-containing compositions (slurry) 1 to 3 were used in the experiments described below.

[2] Preparation of Flavor-Carrying Cigarette Constituent Member

[2-1] Preparation of Flavor-Carrying Cut Tobacco

[0056] The menthol-containing composition (slurry) (20 parts by weight) (about 60°C, in the state of a sol) was uniformly applied to cut tobacco of Mild Seven Super Lights product (Japan Tobacco Inc.) (100 parts by weight). In this experiment, the menthol-containing composition (slurry) was added to the cut tobacco using a transfer pump (Heisin Mohno-Pump 2NVL06F manufactured by HEISHIN Ltd.).

[0057] The cut tobacco to which the menthol-containing composition (slurry) was added was once cooled by blowing cold air having a temperature of 10°C (cold air generator: Suiden SS-25DD-1) until the temperature of the menthol-containing composition added to the cut tobacco reached 20°C or lower (product temperature-measuring device: PT-7LD manufactured by Optex Co., Ltd.).

[0058] After that, the cooled cut tobacco was dried through a belt conveyor type hot air dryer (hot air temperature conditions: 110°C, 2.5 minutes → 100°C, 5 minutes → 80°C, 2.5 minutes) until the water content of the cut tobacco (to which the menthol-containing composition has been added) reached about 12% by weight (see "Measurement of Water Content" described below). The flocking cut tobacco was disentangled, whereby a "flavor-carrying cut tobacco of the present invention" was prepared. The cut tobacco to which the menthol-containing composition 1 has been applied, is referred to as "flavor-carrying cut tobacco 1 of the present invention". The cut tobacco to which the menthol-containing composition 2 has been applied, is referred to as "flavor-carrying cut tobacco 2 of the present invention". The cut tobacco to which the menthol-containing composition 3 has been applied, is referred to as "flavor-carrying cut tobacco 3 of the present invention". The flavor-carrying cut tobacco 1 to 3 of the present invention all had a water content of 12% by weight to 13% by weight.

[0059] On the other hand, a control sample for the cut tobacco was obtained by adding menthol to cut tobacco in a usual manner. In the control sample, the flavor (menthol) was applied to cut tobacco by spraying an ethanol solution containing the flavor on the cut tobacco. The flavor was applied so that the cut tobacco had a menthol amount of 1.0% by weight per weight of the cut tobacco.

[2-2] Preparation of Flavor-Carrying Sheet Tobacco

[0060] The menthol-containing composition (slurry) (about 60°C, in the state of a sol) was applied to sheet tobacco (a reconstituted tobacco material), which was produced by a paper-making process, as uniformly as possible. Specifically, using a film applicator, the menthol-containing composition (slurry) was casted on the sheet tobacco (0.2 mm-thickness), whereby the sheet tobacco was coated with the menthol-containing composition (slurry). Here, the menthol-containing composition (slurry) was coated in an amount of 150 parts by weight based on the sheet tobacco (100 parts by weight).

[0061] The sheet tobacco coated with the menthol-containing composition (slurry) was once cooled by blowing cold air having a temperature of 10°C (cold air generator: Suiden SS-25DD-1) until the temperature of the menthol-containing composition coated on the sheet tobacco reached 20°C or lower (product temperature-measuring device: PT-7LD manufactured by Optex Co., Ltd.).

[0062] After that, the cooled sheet tobacco was dried through a belt conveyor type hot air dryer (hot air temperature conditions: 110°C, 2.5 minutes → 100°C, 5 minutes → 80°C, 2.5 minutes) until the water content of the sheet tobacco (which has been coated with the menthol-containing composition) reached about 10% by weight (see "Measurement of Water Content" described below), whereby a "flavor-carrying sheet tobacco of the present invention" was prepared. The sheet tobacco to which the menthol-containing composition 1 was applied, is referred to as "flavor-carrying sheet tobacco 1 of the present invention". The sheet tobacco to which the menthol-containing composition 2 was applied, is referred to as "flavor-carrying sheet tobacco 2 of the present invention". The sheet tobacco to which the menthol-containing composition 3 was applied, is referred to as "flavor-carrying sheet tobacco 3 of the present invention". The flavor-carrying sheet tobacco 1 to 3 of the present invention all had a water content of 9% by weight to 10% by weight.

[0063] On the other hand, a control sample for the sheet tobacco was obtained by using a usual flavor application method used in cut tobacco. In the control sample, the flavor (menthol) was applied to sheet tobacco by spraying an ethanol solution containing the flavor on the sheet tobacco. The flavor was applied so that the sheet tobacco had a menthol amount of 5.0% by weight per weight of the sheet tobacco.

[2-3] Preparation of Flavor-Carrying Cigarette Paper

[0064] The menthol-containing composition (slurry) (about 60°C, in the state of a sol) was applied to a cigarette paper (a basis weight of 40 g/m², and a thickness of 35 μm) as uniformly as possible. Specifically, using a film applicator, the menthol-containing composition (slurry) was casted on the cigarette paper to a thickness of 0.05 mm. Here, the menthol-containing composition (slurry) was coated in an amount of 40 parts by weight based on the cigarette paper (100 parts by weight).

[0065] The cigarette paper coated with the menthol-containing composition (slurry) was once cooled by blowing cold air having a temperature of 10°C (cold air generator: Suiden SS-25DD-1) until the temperature of the menthol-containing composition coated on the cigarette paper reached 20°C or lower (product temperature-measuring device: PT-7LD manufactured by Optex Co., Ltd.).

[0066] After that, the cooled cigarette paper was dried through a belt conveyor type hot air dryer (hot air temperature conditions: 110°C, 2.5 minutes → 100°C, 5 minutes → 80°C, 2.5 minutes) until the water content of the cigarette paper (which has been coated with the menthol-containing composition) reached about 10% by weight (see "Measurement of Water Content" described below), whereby a "cigarette paper of the present invention" was prepared.

[0067] The cigarette paper to which the menthol-containing composition 1 was applied, is referred to as "flavor-carrying cigarette paper 1 of the present invention". The cigarette paper to which the menthol-containing composition 2 was applied, is referred to as "flavor-carrying cigarette paper 2 of the present invention". The cigarette paper to which the menthol-containing composition 3 was applied, is referred to as "flavor-carrying cigarette paper 3 of the present invention". The flavor-carrying cigarette papers 1 to 3 of the present invention all had a water content of 6% by weight to 7% by weight.

[0068] On the other hand, a control sample for the cigarette paper was obtained by using a usual flavor application method used in cut tobacco. In the control sample, the flavor (menthol) was applied to the cigarette paper by spraying an ethanol solution containing the flavor on the cigarette paper. The flavor was applied so that the cigarette paper had a menthol amount of 1.0% by weight per weight of the cigarette paper.

[3] Measurement of Water Content

[0069] In order to acquire a dryness state of the menthol-containing composition slurry applied to the cigarette constituent member, a water content was measured according to the following method.

[3-1] Preparation of Solution for Measuring Water Content

[0070] An amount of 0.1 g of the prepared flavor-carrying cigarette constituent member was weighed, to which 10 mL of methanol (a new reagent of special grade or higher grade was dispensed through a dispenser without exposure to the air in order to exclude an influence caused by absorption of water in the air) was added in a 50 mL airtight container (which had been stored in a desiccator). The resulting mixture was shaken in a shaker (SHAKER SRR-2 manufactured by AS ONE) for 40 minutes (linear reciprocating shaking (reciprocating), 3 cm-amplitude, 200 revolutions per minute). It was allowed to stand overnight, and then the mixture was shaken again for 40 minutes (the same conditions as above: 200 rpm). The supernatant obtained after allowing it to stand (it was exposed to as little air as possible) was filled in a vial, which was used as a solution for measuring a water content.

[3-2] GC-TCD Water Content Measurement

[0071] The obtained solution for measuring a water content was subjected to a GC-TCD as described below, and quantified according to a calibration curve method.

GC-TCD: 6890 Gas Chromatograph manufactured by Hewlett Packard
Column: HP Polapack Q (packed column)
Carrier Gas: He

Constant Flow mode 20.0 mL/minute

Injection: 1.0 μL
Inlet: EPC purge packed column inlet

Heater: 230°C
Inlet Gas: He
Total flow: 21.1 mL/minute

EP 2 891 409 A1

Oven: 160°C (hold 4.5 minutes) → (60°C/minute) → 220°C (hold 4.0 minutes)

Detector: TCD detector

Reference Gas (He) flow rate: 20 mL/minute

Make up gas (He) 3.0 mL/minute

Signal rate: 5 Hz

Concentration of calibration curve solution: six points of 0, 1, 3, 5, 10 and 20 [mg-H₂O/10 mL]

[4] Measurement of Menthol Content

[0072] A "menthol content of a flavor-carrying cigarette constituent member" and a "menthol content in mainstream smoke" were measured according to the following methods.

[0073] The menthol content of the prepared flavor-carrying cigarette constituent member was measured in order to examine a post-storage flavor-retaining property and a blending uniformity of a flavor. In addition, the menthol content in the mainstream smoke was measured in order to examine a post-storage flavor-retaining property.

[4-1] Preparation of Solution for Measuring Menthol Content of Cigarette Constituent Member

[0074] An amount of 0.1 g of the prepared flavor-carrying cigarette constituent member was weighed, to which 10 mL of methanol (a reagent of special grade or higher grade) was added in a 50 mL airtight container (a screw tube). The resulting mixture was shaken in a shaker (SHAKER SRR-2 manufactured by AS ONE) for 40 minutes (linear reciprocating shake (reciprocating), 3 cm-amplitude, 200 revolutions per minute). It was allowed to stand overnight, and then the mixture was shaken again for 40 minutes (the same conditions as above: 200 rpm). The supernatant obtained after allowing it to stand was diluted at an appropriate dilution degree suitable for a GC measurement (here, x10 dilution with methanol), which was used as a solution for measuring menthol to be subjected to the GC-FID measurement.

[4-2] Preparation of Solution for Measuring Menthol Content in Mainstream Smoke

[0075] An extraction solution from a Cambridge filter was prepared as described below, which was used as a solution for measuring menthol. Specifically, 10 mL of methanol (a reagent of special grade or higher grade) was added to a Cambridge filter sample in a 50 mL airtight container (a screw tube), and the mixture was shaken in a shaker (SHAKER SRR-2 manufactured by AS ONE) for 40 minutes (linear reciprocating shake (reciprocating), 3 cm-amplitude, 200 revolutions per minute). The supernatant obtained after allowing it to stand was filled in a vial, which was used as a solution for measuring menthol for being subjected to a GC-FID measurement.

[4-3] GC-FID Menthol Content Measurement

[0076] The obtained solution for measuring menthol was subjected to a GC-FID as described below, and quantified according to a calibration curve method.

GC-FID: 6890N Gas chromatograph manufactured by Agilent Technologies, Inc.

Column: DB-WAX 30 m x 530 μm x 1 μm [Max 220°C]

Carrier Gas: He

Constant Pressure mode 5.5 psi (velocity: 50 cm/second)

Injection: 1.0 μL

Inlet: Splitless mode 250°C 5.5 psi

Oven: 80°C → (10°C/minute) → 170°C (hold 6.0 minutes)

Detector: FID detector 250°C (H₂: 40 mL/minute-air: 450 mL/minute)

Signal rate: 20 Hz

Concentration of calibration curve solution: eight points of 0, 0.01, 0.05, 0.1, 0.3, 0.5, 0.7 and 1.0 [mg-menthol/mL]

[5] Acceleration Test

[0077] The prepared flavor-carrying cigarette constituent member was put in a thermostat set at a temperature of 50°C (DX 600 manufactured by Yamato Scientific Co., Ltd.), which was stored over one week, two weeks or one month (30

days). A menthol content was measured at every time point as described above, and the post-storage flavor-retaining property was evaluated based on the change of the menthol content.

[6] Production of Cigarette and Smoking Experiment

[0078] Using the flavor-carrying cigarette constituent member of the present invention, a cigarette was produced as described below.

[0079] A cigarette was prepared in conformity with Mild Seven Super Lights (Japan Tobacco Inc.) in which each of the prepared flavor-carrying cigarette constituent members was used.

[0080] Specifically, a cigarette comprising the flavor-carrying cut tobacco of the present invention was produced by using cut tobacco of a Mild Seven Super Lights product as a starting material, preparing the flavor-carrying cut tobacco of the present invention, and using it together with various material products (a cigarette paper, a filter, and the like) for producing the Mild Seven Super Lights.

[0081] A cigarette comprising the flavor-carrying sheet tobacco of the present invention was produced by blending the cut tobacco (85 parts by weight) for producing Mild Seven Super Lights with the flavor-carrying sheet tobacco of the present invention (15 parts by weight), and using it in a predetermined filling amount together with various material products (a cigarette paper, a filter, and the like) for producing the Mild Seven Super Lights product.

[0082] A cigarette comprising the flavor-carrying cigarette paper of the present invention was produced by replacing a cigarette paper of the Mild Seven Super Lights product with the flavor-carrying cigarette paper of the present invention. The cigarettes were produced so that tar values were organized as uniformly as possible.

[0083] A smoking experiment was performed in standard smoking conditions as prescribed in ISO (puff duration of 2 seconds (suspending duration of 58 seconds) in one minute, puff volume of 35 mL, and cigarette butt length of 35 mm (tipping paper + 3 mm)).

[0084] Smoke collected from the Cambridge filter was extracted as described above, and a menthol content in the mainstream smoke was measured as described above.

[7] Results (Cut Tobacco)

[0085] With respect to the cut tobacco, the following results were obtained using the experimental methods described above.

[7-1] Post-storage Flavor-Retaining Property of Menthol (Evaluation in a case of using the cut tobacco)

[0086] Using the "flavor-carrying cut tobacco 1 to 3 of the present invention" and the "control sample of cut tobacco" prepared as described above, the post-storage flavor-retaining property of menthol in the cut tobacco was evaluated. The results are shown in FIG. 1.

[0087] The cut tobacco was allowed to stand for a predetermined period (0 days, 7 days, 14 days, or 30 days) in an acceleration environment. The menthol content of the cut tobacco was measured according to the GC-FID measurement as described above. In FIG. 1, the results of the flavor-carrying cut tobacco 1 of the present invention, the results of the flavor-carrying cut tobacco 2 of the present invention, the results of the flavor-carrying cut tobacco 3 of the present invention, and the results of the control sample are respectively shown as "Carrageenan", "Gellan gum", "Gellan gum + tamarind gum" and "Control". The menthol content (% by weight) is obtained from a formula: {measured value of menthol content (mg)/weight of cut tobacco (mg)} x 100.

[0088] The control sample had a menthol content of about 0.7% by weight at a time point when the storage was started (day 0). When the control sample was stored in a thermostat set at a temperature of 50°C, the menthol content showed a tendency to be remarkably reduced with the lapse of time.

[0089] In contrast, the flavor-carrying cut tobacco 1 to 3 of the present invention had menthol contents of 1.2 to 1.5% by weight at a time point when the storage was started (day 0), and they showed the good flavor-retaining property for at least one month even after the storage at 50°C in the acceleration environment.

[7-2] Post-storage Flavor-Retaining Property of Menthol (Evaluation in a case of using the cigarette)

[0090] Using the cigarettes comprising the "flavor-carrying cut tobacco 1 to 3 of the present invention" and the control cigarette comprising the "control sample of cut tobacco", the post-storage flavor-retaining property of menthol in the cigarettes was evaluated. The results are shown in FIG. 2.

[0091] The cut tobacco was allowed to stand for a predetermined period (0 days, 7 days, 14 days, or 30 days) in an acceleration environment as described above, and then cigarettes were produced using the cut tobacco. The smoking experiment was performed using the cigarettes. The menthol content in mainstream smoke was measured according

to the GC-FID measurement as described above.

[0092] In FIG. 2, the results of the cigarette comprising the flavor-carrying cut tobacco 1 of the present invention, the results of the cigarette comprising the flavor-carrying cut tobacco 2 of the present invention, the results of the cigarette comprising the flavor-carrying cut tobacco 3 of the present invention, and the results of the cigarette comprising the control sample are respectively shown as "Carrageenan", "Gellan gum", "Gellan gum + tamarind gum" and "Control". In FIG. 2, the menthol amount in mainstream smoke [mg/cig] shows a measured menthol amount (mg) per cigarette.

[0093] The cigarette comprising the control sample of cut tobacco showed a large menthol amount in the mainstream smoke when the cut tobacco stored for 0 days was used, but the menthol amount was remarkably reduced when the cut tobacco stored for 7 days was used.

[0094] In contrast, the cigarettes comprising the flavor-carrying cut tobacco 1 to 3 of the present invention were observed to have the good flavor-retaining property for at least one month even if the cut tobacco, which was stored at 50°C in the acceleration environment, was used.

[7-3] Blending Uniformity of Menthol

[0095] With respect to the 10 cigarettes comprising the flavor-carrying cut tobacco of the present invention, the extent of the variation in the blending amount of the flavor among the cigarettes was examined. In addition, the cigarette rod comprising the flavor-carrying cut tobacco of the present invention was divided into three parts, and the extent of the variation in the blending amount of the flavor among the parts was also examined. The results are shown in Table 1, in which the former is shown in "Variation between cigarettes" and the latter is shown in "Variation in cigarette". "Variation in cigarette (CV%)" is represented by an average value of the measurements of the 10 cigarettes.

[0096] With respect to the control, a sample cigarette was produced by blending 2% by weight of cut pieces of a menthol-containing sheet (WO 2009/142159), instead of the flavor-carrying cut tobacco of the present invention, with cut tobacco, and making a tar value uniform. The menthol amount in the mainstream smoke of this sample cigarette was 0.4 mg per cigarette, which was almost equal to that obtained in the cigarette using the flavor-carrying cut tobacco of the present invention.

[Table 1]

Cut tobacco	Variation between cigarettes [CV%]	Variation in cigarette [CV%]
Control (Cut pieces of menthol sheet)	34%	54%
Flavor-carrying cut tobacco 1 of present invention	14%	15%
Flavor-carrying cut tobacco 2 of present invention	14%	14%
Flavor-carrying cut tobacco 3 of present invention	13%	15%

[0097] In the case where the menthol-containing sheet was cut into the same shape as the cut tobacco and the resulting cut sheet was blended with the cut tobacco, the variation between the cigarettes (CV%) was 34%, and the variation in the cigarette was 54%. This variation was caused by the low blend content of the menthol-containing sheet, and if the blend content of the menthol-containing sheet is 10% by weight or more, the variation (CV%) reduces to about 10%. On the other hand, when the cigarette was prepared using the flavor-carrying cut tobacco of the present invention, both the variation between the cigarettes and the variation in the cigarette were remarkably reduced compared to those of the control.

[0098] From those results, it is found that the cigarette comprising the flavor-carrying cut tobacco of the present invention can better suppress the variation in the blending amount of menthol between the cigarettes and more uniformly blend menthol in the cigarette rod than the cigarette obtained by blending cut pieces of the menthol-containing sheet as the tobacco filler. The cigarette comprising the flavor-carrying cut tobacco of the present invention, accordingly, can suppress the variation in the smoking flavor and taste for each cigarette, and the variation in the smoking flavor and taste over the cigarette smoking time. In addition, in the present invention, the flavor composition is applied to a commonly used cigarette constituent member, and thus the cigarette manufacturing can be easily performed in existing facilities.

[8] Results (Sheet Tobacco)

[0099] With respect to the sheet tobacco, the following results were obtained using the experimental methods described above.

[8-1] Post-storage Flavor-Retaining Property of Menthol (Evaluation in a case of using the sheet tobacco)

[0100] Using the "flavor-carrying sheet tobacco 1 to 3 of the present invention" and the "control sample of sheet tobacco" prepared as described above, the post-storage flavor-retaining property of menthol in the sheet tobacco was evaluated. The results are shown in FIG. 3.

[0101] The sheet tobacco was allowed to stand for a predetermined period (0 days, 7 days, 14 days, or 30 days) in an acceleration environment. The menthol content of the sheet tobacco was measured according to the GC-FID measurement as described above. In FIG. 3, the results of the flavor-carrying sheet tobacco 1 of the present invention, the results of the flavor-carrying sheet tobacco 2 of the present invention, the results of the flavor-carrying sheet tobacco 3 of the present invention, and the results of the control sample are respectively shown as "Carrageenan", "Gellan gum", "Gellan gum + tamarind gum" and "Control". The menthol content (% by weight) is obtained from a formula: {measured value of menthol content (mg)/weight of sheet tobacco (mg)} x 100.

[0102] The control sample had a menthol content of about 3% by weight at a time point when the storage was started (day 0). When the control sample was stored in a thermostat set at a temperature of 50°C, the menthol content was remarkably reduced with the lapse of time and it reached almost 0 after one week had elapsed.

[0103] In contrast, the flavor-carrying sheet tobacco 1 to 3 of the present invention had menthol contents of about 5 to 7% by weight at a time point when the storage was started (day 0), and they showed comparatively high menthol contents even after the storage at 50°C in the acceleration environment; i.e., the good flavor-retaining property was observed.

[8-2] Post-storage Flavor-Retaining Property of Menthol (Evaluation in a case of using the cigarette)

[0104] Using the cigarettes comprising the "flavor-carrying sheet tobacco 1 to 3 of the present invention" and the control cigarette comprising the "control sample of sheet tobacco", the post-storage flavor-retaining property of menthol in the cigarettes was evaluated. The results are shown in FIG. 4.

[0105] The sheet tobacco was allowed to stand for a predetermined period (0 days, 7 days, 14 days, or 30 days) in an acceleration environment as described above, and then cigarettes were produced using the sheet tobacco. The menthol content in mainstream smoke was measured according to the GC-FID measurement as described above.

[0106] In FIG. 4, the results of the cigarette comprising the flavor-carrying sheet tobacco 1 of the present invention, the results of the cigarette comprising the flavor-carrying sheet tobacco 2 of the present invention, the results of the cigarette comprising the flavor-carrying sheet tobacco 3 of the present invention, and the results of the cigarette comprising the control sample are respectively shown as "Carrageenan", "Gellan gum", "Gellan gum + tamarind gum" and "Control". In FIG. 4, the menthol amount in mainstream smoke [mg/cig] shows a measured menthol amount (mg) per cigarette.

[0107] The cigarette comprising the control sample of sheet tobacco showed a menthol amount in the mainstream smoke which is equal to that in the cigarette comprising the flavor-carrying sheet tobacco of the present invention, when the sheet tobacco stored for 0 days was used, but the menthol amount was almost 0 when the sheet tobacco stored for 7 days was used.

[0108] In contrast, the cigarette samples comprising the flavor-carrying sheet tobacco 1 to 3 had comparatively large menthol amounts in the mainstream smoke, and were observed to have the good flavor-retaining property, even if the sheet tobacco, which was stored at 50°C in the acceleration environment, was used.

[8-3] Blending Uniformity of Menthol

[0109] With respect to the 10 cigarettes comprising the flavor-carrying sheet tobacco of the present invention, the extent of the variation in the blending amount of the flavor among the cigarettes was examined. In addition, the cigarette rod comprising the flavor-carrying sheet tobacco of the present invention was divided into three parts, and the extent of the variation in the blending amount of the flavor among the parts was also examined. The results are shown in Table 2, in which the former is shown in "Variation between cigarettes" and the latter is shown in "Variation in cigarette". "Variation in cigarette (CV%)" is represented by an average value of the measurements of the 10 cigarettes.

[0110] With respect to the control, a sample cigarette was produced by blending 2% by weight of cut pieces of a menthol-containing sheet (WO 2009/142159) with cut tobacco, and making a tar value uniform, in the same manner as used in the item of the flavor-carrying cut tobacco described above.

[Table 2]

Sheet tobacco	Variation between cigarettes [CV%]	Variation in cigarette [CV%]
Control (Cut pieces of menthol sheet)	34%	54%

(continued)

Sheet tobacco	Variation between cigarettes [CV%]	Variation in cigarette [CV%]
Flavor-carrying sheet tobacco 1 of present invention	15%	15%
Flavor-carrying sheet tobacco 2 of present invention	15%	16%
Flavor-carrying sheet tobacco 3 of present invention	14%	16%

[0111] In the case where the menthol-containing sheet was cut into the same shape as the cut tobacco and the resulting cut sheet (2% by weight) was blended with the cut tobacco, the variation between the cigarettes (CV%) was 34%, and the variation in the cigarette was 54%. On the other hand, when the cigarette was prepared using the flavor-carrying sheet tobacco of the present invention (the blended amount is 15% by weight based on the total weight), both the variation between the cigarettes and the variation in the cigarette were remarkably reduced compared to those of the control.

[0112] From those results, it is found that the cigarette comprising the flavor-carrying sheet tobacco of the present invention can better suppress the variation in the blending amount of menthol between the cigarettes and more uniformly blend menthol in the cigarette rod than the cigarette obtained by blending cut pieces of the menthol-containing sheet as the tobacco filler. The cigarette comprising the flavor-carrying sheet tobacco of the present invention, accordingly, can suppress the variation in the smoking flavor and taste for each cigarette, and the variation in the smoking flavor and taste over the cigarette smoking time.

[9] Results (Cigarette Paper)

[0113] With respect to the cigarette paper, the following results were obtained using the experimental methods described above.

[9-1] Post-storage Flavor-Retaining Property of Menthol (Evaluation in a case of using the cigarette paper)

[0114] Using the "flavor-carrying cigarette papers 1 to 3 of the present invention" and the "control sample of cigarette paper" prepared as described above, the post-storage flavor-retaining property of menthol in the cigarette paper was evaluated. The results are shown in FIG. 5.

[0115] The cigarette paper was allowed to stand for a predetermined period (0 days, 7 days, 14 days, or 30 days) in an acceleration environment. The menthol content of the cigarette paper was measured according to the GC-FID measurement as described above. In FIG. 5, the results of the flavor-carrying cigarette paper 1 of the present invention, the results of the flavor-carrying cigarette paper 2 of the present invention, the results of the flavor-carrying cigarette paper 3 of the present invention, and the results of the control sample are respectively shown as "Carrageenan", "Gellan gum", "Gellan gum + tamarind gum" and "Control". The menthol content (% by weight) is obtained from a formula: {measured value of menthol content (mg)/weight of cigarette paper (mg)} x 100.

[0116] The control sample had a menthol content of about 0.7% by weight at a time point when the storage was started (day 0). When the control sample was stored in a thermostat set at a temperature of 50°C, the menthol content was remarkably reduced with the lapse of time and it reached almost 0 after one week had elapsed.

[0117] In contrast, the flavor-carrying cigarette papers 1 to 3 of the present invention had menthol contents of about 1.4 to 1.7% by weight at a time point when the storage was started (day 0), and they showed comparatively high menthol contents even after the storage at 50°C in the acceleration environment; i.e., the good flavor-retaining property was observed.

[9-2] Post-storage Flavor-Retaining Property of Menthol (Evaluation in a case of using the cigarette)

[0118] Using the cigarettes comprising the "flavor-carrying cigarette papers 1 to 3 of the present invention" and the control cigarette comprising the "control sample of cigarette paper", the post-storage flavor-retaining property of menthol in the cigarette was evaluated. The results are shown in FIG. 6.

[0119] The cigarette paper was allowed to stand for a predetermined period (0 days, 7 days, 14 days, or 30 days) in an acceleration environment as described above, and then cigarettes were produced using the cigarette paper as

described above. The menthol content in mainstream smoke was measured according to the GC-FID measurement as described above.

[0120] In FIG. 6, the results of the cigarette comprising the flavor-carrying cigarette paper 1 of the present invention, the results of the cigarette comprising the flavor-carrying cigarette paper 2 of the present invention, the results of the cigarette comprising the flavor-carrying cigarette paper 3 of the present invention, and the results of the cigarette comprising the control sample are respectively shown as "Carrageenan", "Gellan gum", "Gellan gum + tamarind gum" and "Control". In FIG. 6, the menthol amount in mainstream smoke [mg/cig] shows a measured menthol amount (mg) per cigarette.

[0121] The cigarette comprising the control sample of cigarette paper showed a menthol amount in the mainstream smoke which is equal to that in the cigarette comprising the flavor-carrying cigarette paper of the present invention, when the cigarette paper stored for 0 day was used, but the menthol amount was almost 0 when the cigarette paper stored for 7 days was used.

[0122] In contrast, the cigarettes comprising the flavor-carrying cigarette papers 1 to 3 had comparatively large menthol amounts in the mainstream smoke, and were observed to have the good flavor-retaining property, even if the cigarette paper, which was stored at 50°C in the acceleration environment, was used.

[9-3] Blending Uniformity of Menthol

[0123] With respect to the 10 cigarettes comprising the flavor-carrying cigarette paper of the present invention, the extent of the variation in the blending amount of the flavor among the cigarettes was examined. In addition, the cigarette rod comprising the flavor-carrying cigarette paper of the present invention was divided into three parts, and the extent of the variation in the blending amount of the flavor among the parts was also examined. The results are shown in Table 3, in which the former is shown in "Variation between cigarettes" and the latter is shown in "Variation in cigarette". "Variation in cigarette (CV%)" is represented by an average value of the measurements of the 10 cigarettes.

[0124] With respect to the control, a sample cigarette was produced by blending 2% by weight of cut pieces of a menthol-containing sheet (WO 2009/142159) with cut tobacco, and making a tar value uniform, in the same manner as used in the item of the flavor-carrying cut tobacco described above.

[Table 3]

Cigarette paper	Variation between cigarettes [CV%]	Variation in cigarette [CV%]
Control (Cut pieces of menthol sheet)	34%	54%
Flavor-carrying cigarette paper 1 of present invention	9%	7%
Flavor-carrying cigarette paper 2 of present invention	11%	10%
Flavor-carrying cigarette paper 3 of present invention	12%	9%

[0125] In the case where the menthol-containing sheet was cut into the same shape as the cut tobacco and the resulting cut sheet (2% by weight) was blended with the cut tobacco, the variation between the cigarettes (CV%) was 34%, and the variation in the cigarette was 54%. On the other hand, when the cigarette was prepared using the flavor-carrying cigarette paper of the present invention, both the variation between the cigarettes and the variation in the cigarette were remarkably reduced compared to those of the control.

[0126] From those results, it is found that the cigarette comprising the flavor-carrying cigarette paper of the present invention can better suppress the variation in the blending amount of menthol between the cigarettes and more uniformly blend menthol in the cigarette than the cigarette obtained by blending cut pieces of the menthol-containing sheet as the tobacco filler. The cigarette comprising the flavor-carrying cigarette paper of the present invention, accordingly, can suppress the variation in the smoking flavor and taste for each cigarette, and the variation in the smoking flavor and taste over the cigarette smoking time.

[10] Temperature-Responsive Sol-Gel Transition Property of Polysaccharide-Containing Liquid

[0127] In this experiment, a temperature-responsive sol-gel transition property was examined for the following polysaccharide-containing liquid (slurry) (1) to (3).

[10-1] Preparation of Polysaccharide-Containing Liquid

(1) Carrageenan-Containing Liquid

[0128]

Water 0.1 liters
κ-Carrageenan (CARRAGEENAN CS-530/San-Ei Gen F.F.I., Inc.) 5 grams

(2) Gellan Gum-Containing Liquid

[0129]

Water 0.1 liters
Gellan Gum (KELCOGEL/San-Ei Gen F.F.I., Inc.) 5 grams

[0130] The carrageenan-containing liquid and the gellan gum-containing liquid were prepared as described below.

[0131] Water (0.1 L) was kept at 70°C, and the polysaccharide (5 g) was dissolved in the water little by little so as not to form lumps, while the mixture was stirred using a high performance mixer DMM, manufactured by Atec Japan Ltd to prepare the polysaccharide-containing liquid (slurry).

(3) Liquid Containing Gellan Gum and Tamarind Gum

[0132]

Water 10 liters
Gellan Gum (KELCOGEL/San-Ei Gen F.F.I., Inc.) 250 grams
Tamarind Gum (VIS TOP D-2032/San-Ei Gen F.F.I., Inc.) 250 grams
5% Aqueous Lecithin Solution (Sun Lecithin A-1/Taiyo Kagaku Co., Ltd.) 200 milliliters
Menthol (Takasago International Corporation) 2500 grams

[0133] The liquid containing gellan gum and tamarind gum was prepared as described below.

[0134] While 10 liters (100 parts by weight) of water (which was heated to and kept at 80°C) was stirred in a mixer (PRIMIX T. K. AUTO MIXER Model 40/equipped with a solution-stirring rotor/2000 rpm), 500 grams (5 parts by weight) of the two polysaccharides, gellan gum and tamarind gum, in the mixture ratio (weight ratio) described above, were dissolved in the water little by little so as not to form lumps (the time required was about 20 minutes). While the temperature was kept the same, 2500 grams (25 parts by weight) of l-menthol was added thereto. The stirring mixer was changed to a homogenizer (PRIMIX T. K. AUTO MIXER Model 40/equipped with a rotor stator head/4000 rpm) and the mixture was emulsified for 10 minutes. Then, 200 milliliters (2 parts by weight) of the 5% aqueous lecithin solution was added thereto, and the mixture was stirred. Menthol was dispersed in a mixed polysaccharide aqueous solution containing gellan gum and tamarind gum.

[10-2] Results

[0135] The temperature of the polysaccharide-containing liquid (70°C) was dropped to 25°C for about 900 seconds (0.05°C/seconds). After that, the temperature was raised to 70°C for about 900 seconds. How the viscosity (fluidity) of the polysaccharide-containing liquid changed by the temperature variation described above was measured using a rheometer (RheoStress 1 manufactured by Thermo-Haake Ltd.).

[0136] The results of the carrageenan-containing liquid are shown in FIGS. 7A and 7B, the results of the gellan gum-containing liquid are shown in FIGS. 8A and 8B, and the results of the liquid containing gellan gum and tamarind gum are shown in FIGS. 9A and 9B.

[0137] As shown in FIG. 7A, when the temperature of the carrageenan-containing liquid was dropped to 25°C, the viscosity was low until the temperature reached about 50°C, which is the sol-gel transition temperature, but the viscosity was rapidly increased at temperatures lower than the transition temperature (gelation). When the temperature of the obtained gel was raised, the gel did not easily return to sol even if the gel was heated to a temperature higher than the transition temperature, as shown in FIG. 7B. Thus, the gel state could be maintained.

[0138] As shown in FIG. 8A, when the temperature of the gellan gum-containing liquid was dropped to 25°C, the viscosity was low until the temperature reached about 50°C (the fluidity was high), but the viscosity was rapidly increased

at 40°C or lower (gelation phenomenon). When the temperature of the obtained gel was raised, the gel did not easily return to sol even if the gel was heated to a temperature higher than the gelation temperature (40°C), as shown in FIG. 8B. Thus, the gel state could be maintained until the temperature became quite high.

[0139] As shown in FIG. 9A, when the temperature of the liquid containing gellan gum and tamarind gum was dropped to 25°C, the viscosity was low until the temperature reached about 40°C, but the viscosity was rapidly increased at 35°C or lower (gelation phenomenon). When the temperature of the obtained gel was raised, the gel did not easily return to sol even if the gel was heated to a temperature higher than the gelation temperature (35°C), as shown in FIG. 9B. Thus, the gel state could be maintained until the temperature became quite high.

[0140] From those results, it is found that when the slurry containing the polysaccharide described above was once cooled to cause gelation, the gel does not easily return to sol even if the temperature is raised after that, and the gel state can be maintained. Utilizing such a property of the polysaccharide in the present invention, the raw material slurry is subjected to the preliminary cooling before it is dried. As a result, it is difficult to cause the solation of the polysaccharide contained in the raw material slurry which has been subjected to the preliminary cooling, even if the temperature of the slurry is raised at the time of drying, and thus it is difficult to volatilize the menthol coated with such a polysaccharide.

Claims

1. A flavor-carrying cigarette constituent member, which is obtained by:

applying a flavor composition in a sol state to a surface of a cigarette constituent member, the flavor composition containing a flavor, water, and a polysaccharide selected from the group consisting of carrageenan, gellan gum, and a combination of gellan gum and tamarind gum; then
cooling the flavor composition-applied cigarette constituent member to a temperature of 0 to 40°C to cause gelation of the flavor composition; and then
heat-drying the flavor composition-applied cigarette constituent member.

2. The flavor-carrying cigarette constituent member according to claim 1, wherein the heat-drying is performed so that the surface temperature of the flavor composition is kept at 70 to 100°C.

3. The flavor-carrying cigarette constituent member according to claim 1 or 2, wherein the cigarette constituent member is a cigarette paper.

4. The flavor-carrying cigarette constituent member according to claim 1 or 2, wherein the cigarette constituent member is a tobacco filler.

5. The flavor-carrying cigarette constituent member according to claim 4, wherein the tobacco filler is cut tobacco.

6. The flavor-carrying cigarette constituent member according to claim 4, wherein the tobacco filler is sheet tobacco.

7. A cigarette comprising the flavor-carrying cigarette constituent member according to any one of claims 1 to 6.

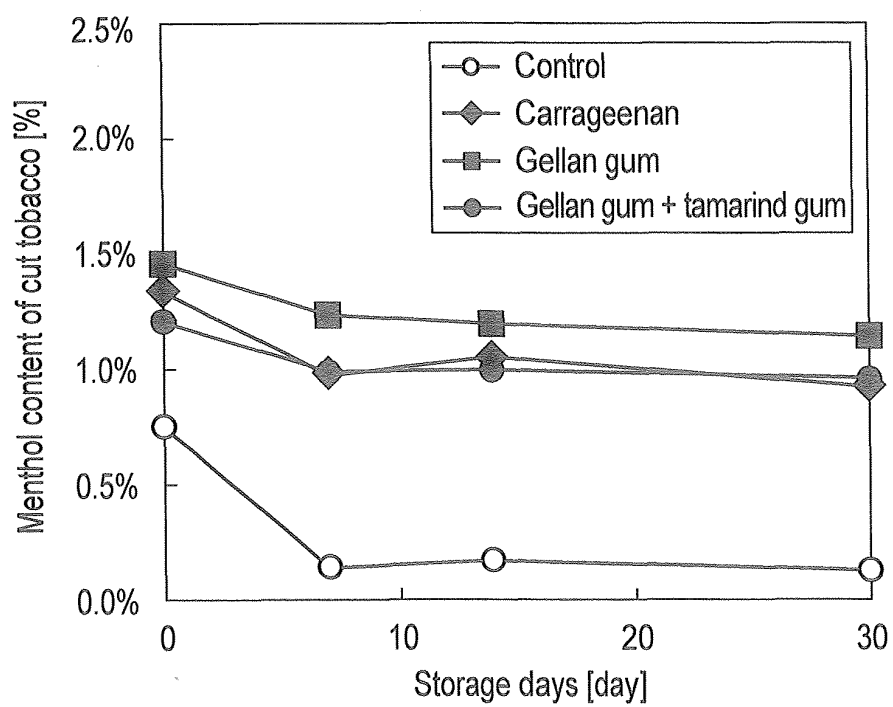


FIG. 1

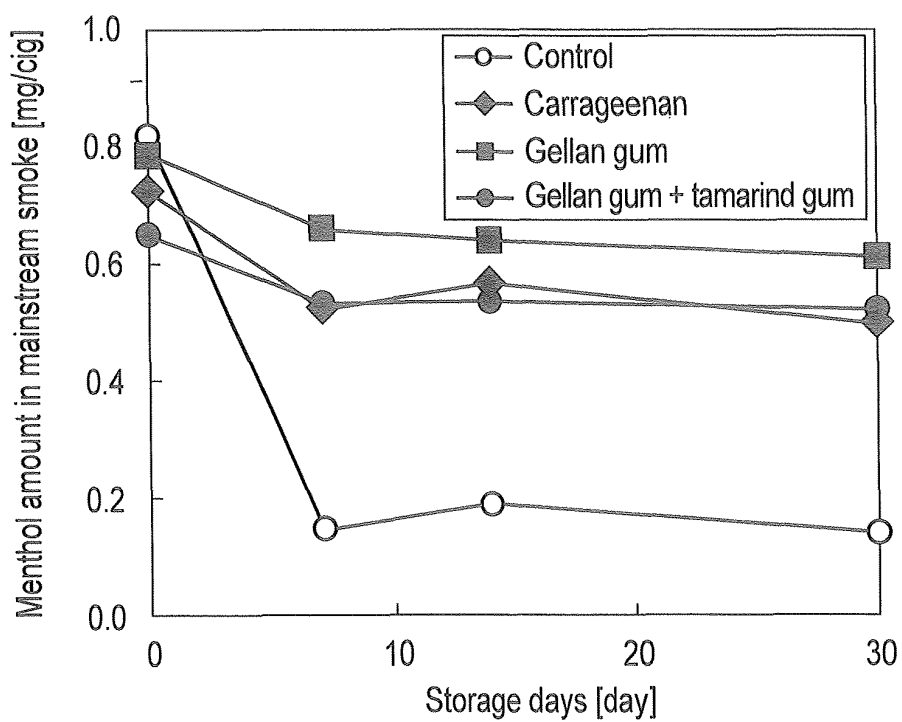


FIG. 2

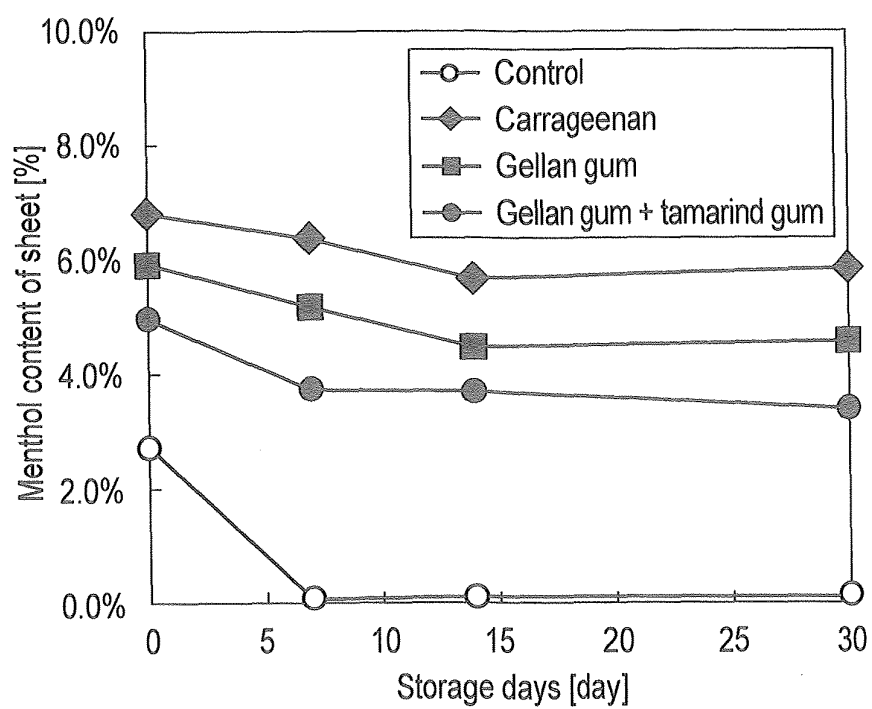


FIG. 3

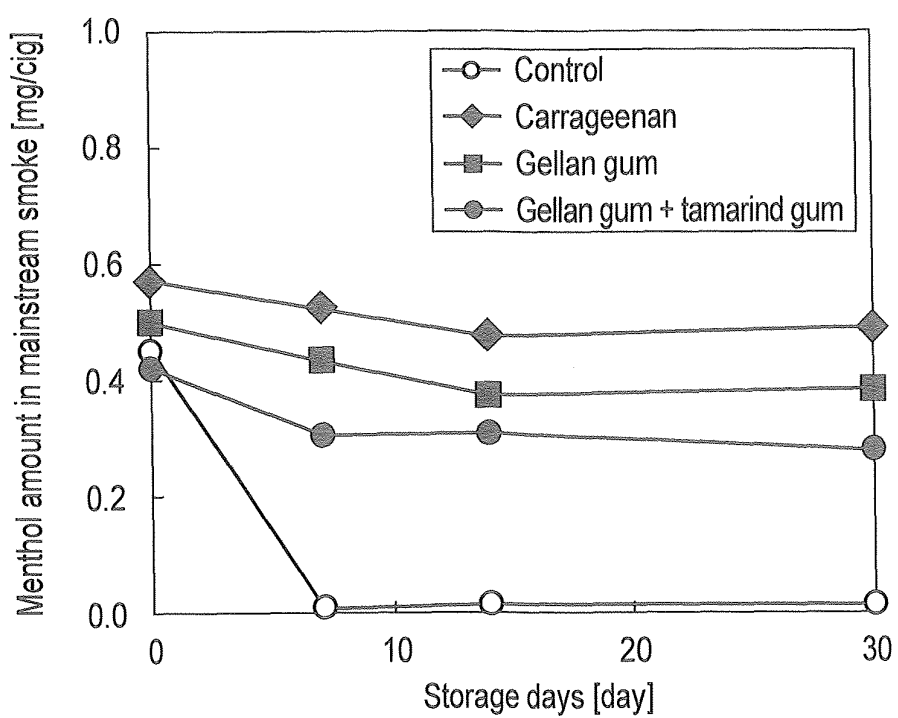


FIG. 4

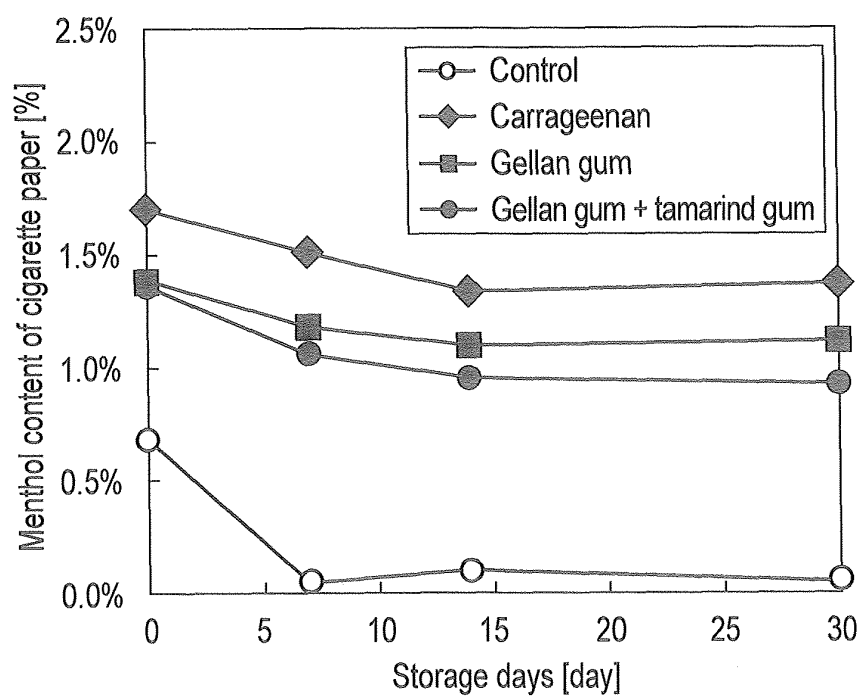


FIG. 5

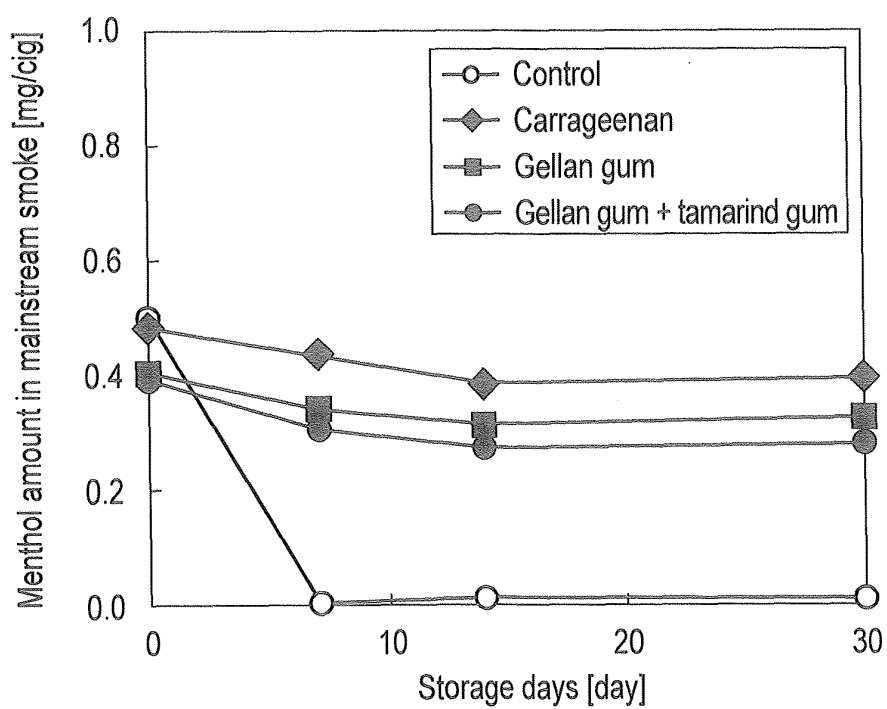


FIG. 6

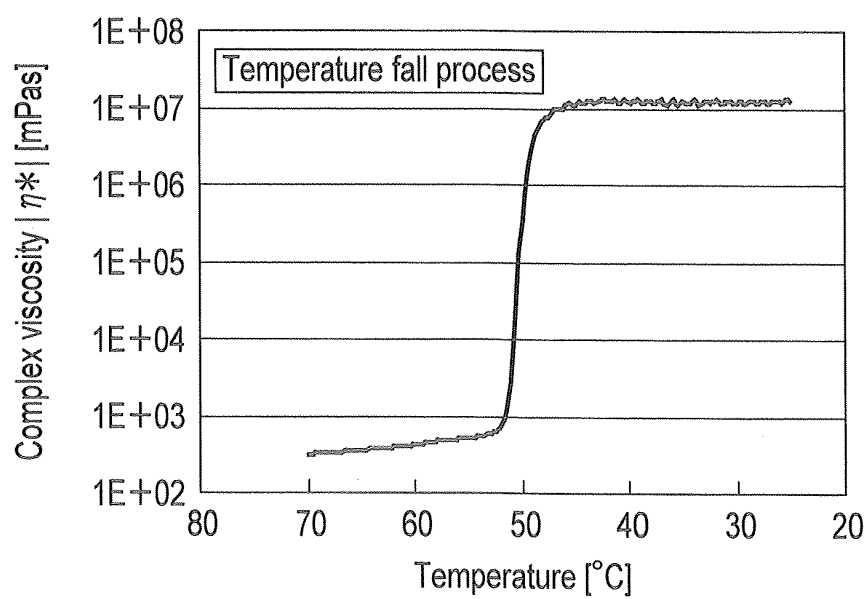


FIG. 7A

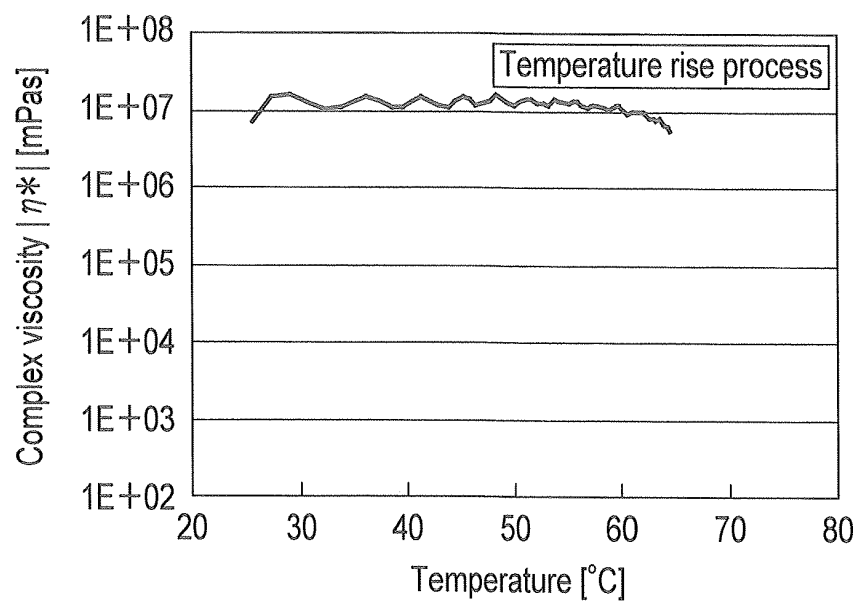


FIG. 7B

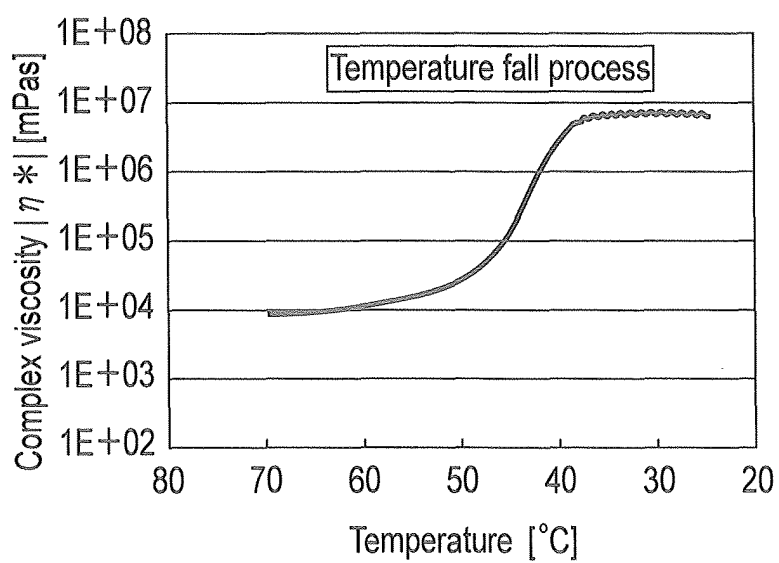


FIG. 8A

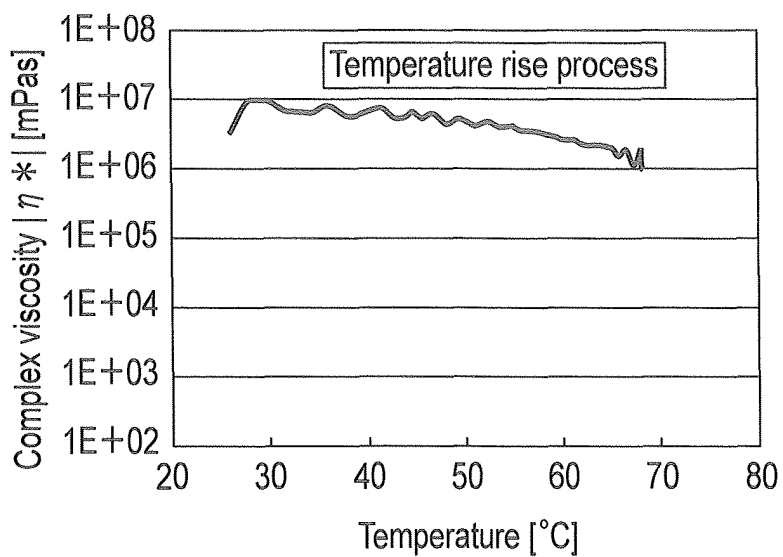


FIG. 8B

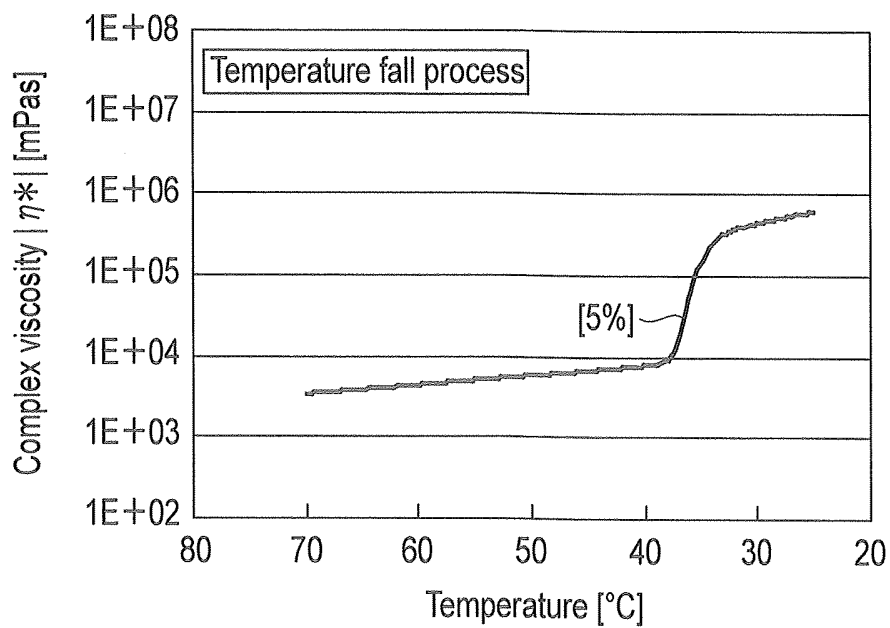


FIG. 9A

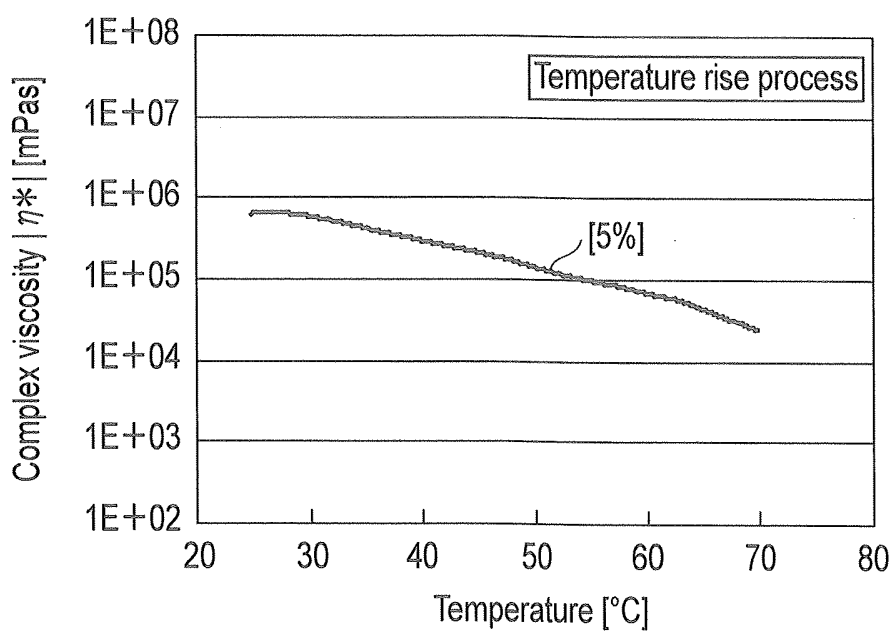


FIG. 9B

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2013/072774

A. CLASSIFICATION OF SUBJECT MATTER

A24B15/30(2006.01)i, A24B3/12(2006.01)i, A24D1/02(2006.01)i, C11B9/00(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)

A24B15/30, A24B3/12, A24D1/02, C11B9/00

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 1996-2013
Kokai Jitsuyo Shinan Koho 1971-2013 Toroku Jitsuyo Shinan Koho 1994-2013

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2009/142159 A1 (Japan Tobacco Inc.), 26 November 2009 (26.11.2009), entire text; all drawings & JP 2013-99346 A & US 2011/0061667 A1 & EP 2279677 A1 & CA 2724820 A & CN 102036575 A & KR 10-2010-0132057 A & TW 201016148 A & RU 2010151986 A	1-7
A	JP 09-028366 A (Japan Tobacco Inc.), 04 February 1997 (04.02.1997), paragraphs [0006] to [0026] (Family: none)	1-7

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

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search
13 November, 2013 (13.11.13)

Date of mailing of the international search report
26 November, 2013 (26.11.13)

Name and mailing address of the ISA/
Japanese Patent Office

Authorized officer

Facsimile No.

Telephone No.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2013/072774

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 11-509566 A (British American Tobacco Co., Ltd.), 24 August 1999 (24.08.1999), entire text & JP 3790828 B & GB 9513951 A & EP 840555 A & WO 1997/002759 A1 & DE 69616332 D & DE 69616332 T & BR 9609628 A & AU 6313996 A & AT 207309 T & DK 840555 T & ES 2162084 T & PT 840555 E & EA 299 B & CN 1193893 A	1-7
A	JP 2007-023065 A (San-Ei Gen F.F.I., Inc.), 01 February 2007 (01.02.2007), paragraphs [0011] to [0021] (Family: none)	1-7
P, X	WO 2012/118032 A1 (Japan Tobacco Inc.), 07 September 2012 (07.09.2012), entire text; all drawings & WO 2012/118033 A & WO 2012/118034 A & TW 201240613 A & TW 201240614 A & TW 201242523 A	1-7

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

REFERENCES CITED IN THE DESCRIPTION

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

Patent documents cited in the description

- JP 9028366 A [0007]
- JP 11509566 W [0007]
- WO 2009142159 A [0007] [0096] [0110] [0124]