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(54) **NICOTINE SUPPLY ORAL POUCH PRODUCT AND PRODUCTION METHOD THEREFOR**

(57) This nicotine supply oral pouch product comprises a composition containing gel particles, and a pouch for packaging the composition. The gel particles contain at least an anionic natural polymer carbohydrate, calcium ions, and water. The composition contains nicotine. The water content of the composition is 15% by weight or more.

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Description

Technical Field

5 **[0001]** The present invention relates to a nicotine supply oral pouch product and a method for producing the nicotine supply oral pouch product.

Background Art

10 **[0002]** Nicotine supply oral pouch products such as oral tobacco products are package bodies in each of which a nicotine-containing composition is housed in a pouch (packaging material) formed of a material such as a nonwoven fabric and are used in the oral cavity of users.

[0003] When a nicotine supply oral pouch product is put into the oral cavity of a user, components such as nicotine in the composition ooze out of the packaging material, whereby an inhaling flavor component is delivered to the user.

15 **[0004]** For nicotine supply oral pouch products, the feeling of use in the oral cavity during use is important, and techniques for improving mouthfeel, techniques for improving affinity between the products and saliva, and other techniques are known. For example, there have been developed a technique in which sealed areas are reduced to decrease the amount of excess air in a product, thereby enabling desired release of flavor during use and improving mouthfeel in the oral cavity (Patent Document 1) and a technique in which certain substances are incorporated in a composition to thereby improve affinity between a product and saliva and promote rapid transmucosal delivery of the substances in the composition in the oral cavity (Patent Document 2).

Citation List

25 Patent Document

[0005]

30 Patent Document 1: Japanese Unexamined Patent Application Publication (Translation of PCT Application) No. 2019-505174

Patent Document 2: Japanese Unexamined Patent Application Publication (Translation of PCT Application) No. 2012-522765

35 Summary of Invention

Technical Problem

40 **[0006]** As described above, in addition to ease of release of substances in a composition and high affinity between a product and saliva, there are other factors that affect the feeling of use in the oral cavity during use of a nicotine supply oral pouch product, and there remains room for improvement in the feeling of use.

45 **[0007]** Regarding typical nicotine supply oral pouch products, the present inventors have focused on the fluidity of a composition in a pouch during use and the adhesion between materials constituting the composition. By increasing the fluidity of a composition in a pouch product, the pouch product can be easily deformed in a direction perpendicular to a direction in which pressure is applied to the pouch product, and thus the user can easily deform the pouch product into a desired shape and hold it in the oral cavity. By decreasing the adhesion between materials constituting the composition, the pouch product can be easily deformed in a direction in which pressure is applied to the pouch product, and thus the user can easily deform the initial pouch product, which has a large thickness, to be thinner.

50 **[0008]** The above improvement in fluidity is an improvement in the feeling of use during the period between when the user puts the pouch product in the mouth and saliva penetrates the pouch product and when the user finishes using the pouch product and takes it out of the mouth, and the above improvement in adhesion is an improvement in the feeling of use at an initial stage from when the user puts the pouch product in the mouth until saliva penetrates the pouch product. Both the improvements in the properties are beneficial to the user. These properties are not disclosed at all in Patent Document 1 or 2 above.

55 **[0009]** Thus, it is an object of the present invention to provide a nicotine supply oral pouch product having improved fluidity or improved adhesion and a method for producing the nicotine supply oral pouch product.

Solution to Problem

[0010] The present inventors have conducted intensive studies and found that the above problems can be solved by using a composition containing gel particles containing specific substances and controlling the water content of the composition to be equal to or above a certain value, thereby accomplishing the present invention.

[1] A nicotine supply oral pouch product includes a composition containing gel particles and a pouch for packaging the composition.

The gel particles contain at least an anionic natural polymer carbohydrate, calcium ions, and water.
The composition contains nicotine.
The water content of the composition is 15% by weight or more.

[2] In the nicotine supply oral pouch product according to [1], the anionic natural polymer carbohydrate has a carboxyl group.

[3] In the nicotine supply oral pouch product according to [2], the anionic natural polymer carbohydrate is LM pectin.

[4] In the nicotine supply oral pouch product according to [2] or [3], in the composition, the ratio of the total number of carboxyl groups of the anionic natural polymer carbohydrate to the total number of the calcium ions is 100:1 to 2:1.

[5] In the nicotine supply oral pouch product according to any one of [1] to [4], a constituent of the composition has a maximum particle size of 15 mm or less when dried.

[6] In the nicotine supply oral pouch product according to any one of [1] to [5], the pouch is a nonwoven fabric.

[7] A method for producing a nicotine supply oral pouch product includes a composition production step of producing a composition containing gel particles comprising at least an anionic natural polymer carbohydrate, a calcium ion supplier, and water.

The composition comprises nicotine.

The water content of the composition is 15% by weight or more.

[8] In the method for producing a nicotine supply oral pouch product according to [7], the calcium ion supplier is a liquid.

[9] In the method for producing a nicotine supply oral pouch product according to [7] or [8], the calcium ion supplier comprises calcium lactate.

Advantageous Effects of Invention

[0011] According to the present invention, a nicotine supply oral pouch product having improved fluidity or improved adhesion and a method for producing the nicotine supply oral pouch product can be provided.

Brief Description of Drawings

[0012]

[Fig. 1] Fig. 1 is a graph showing the relationship between normal stress and shear stress of compositions of Example 2 and Comparative Example 1.

[Fig. 2] Fig. 2 is a graph showing the relationship between normal stress and shear stress of compositions of Example 3 and Comparative Example 2.

[Fig. 3] Fig. 3 is a graph showing the relationship between normal stress and shear stress of compositions of Example 4 and Comparative Example 3.

[Fig. 4] Fig. 4 is a graph showing the relationship between normal stress and shear stress of compositions of Example 1 and Comparative Example 4.

[Fig. 5] Fig. 5 is a graph showing the relationship between normal stress and shear stress of compositions of Example 4 and Comparative Example 5.

[Fig. 6] Fig. 6 is a graph showing the relationship between normal stress and shear stress of compositions of Example 5 and Comparative Example 6.

[Fig. 7] Fig. 7 is a graph showing the relationship between normal stress and shear stress of compositions of Example 6 and Comparative Example 8.

Description of Embodiments

[0013] Although embodiments of the present invention will be described in detail below, these descriptions are examples (representative examples) of embodiments of the present invention, and the present invention is not limited to the contents of the descriptions as long as it does not depart from the spirit thereof.

[0014] In this DESCRIPTION, a numerical range expressed using "to" means a range including numerical values before and after "to" as lower and upper limit values, and "A to B" means A or more and B or less.

<Nicotine Supply Oral Pouch Product>

[0015] A nicotine supply oral pouch product according to an embodiment of the present invention includes a composition containing gel particles and a pouch for packaging the composition.

The gel particles contain at least an anionic natural polymer carbohydrate, calcium ions, and water.

The composition contains nicotine.

The water content of the composition is 15% by weight or more.

[0016] In the oral pouch product, the composition in the pouch contains gel particles, and, furthermore, the gel particles used have a structure in which at least an anionic natural polymer carbohydrate is cross-linked with calcium ions and swollen with a dispersion medium containing water, and the water content of the composition is 15% by weight or more, whereby the fluidity or/and adhesion of the composition can be improved. Specifically, the fluidity or/and adhesion of the anionic natural polymer carbohydrate which swells with water can be improved.

[Composition]

[0017] The composition may have any composition as long as gel particles containing at least an anionic natural polymer carbohydrate, calcium ions, and water, and nicotine are contained. The composition in the present invention is a general term applied to substances contained in the pouch. From the viewpoint of preventing the composition from leaking out of the pouch, the composition is preferably not a liquid, and is preferably formed, for example, of gelatinous gel particles alone or of gel particles and a solid substance. To satisfy a preferred particle size described later, the composition preferably takes a particular shape (a plurality of particles) after being dried.

[0018] The oral pouch product includes a composition containing gel particles. The gel particles contained in the composition are not particularly limited as long as they are in the form of a particulate gel.

[0019] In this DESCRIPTION, the term "particulate" refers to a small particle shape and need not be a perfect sphere, and specifically includes spherical shapes, ellipsoidal shapes, rod-like shapes, plate-like shapes, and shapes apparently close to these shapes.

[0020] In this DESCRIPTION, the term "gelatinous" refers to a state in which a sol-like decomposition product having fluidity is solidified to lose spontaneous fluidity while maintaining elasticity. A substance in such a state is referred to as a "gel", and a substance formed of a gel is referred to as a "gel substance".

[0021] The content of the gel particles in the composition is not particularly limited, and is typically 0.01% by weight or more, preferably 0.05% by weight or more, more preferably 0.1% by weight or more, still more preferably 1.0% by weight or more, and typically 50.0% by weight or less, preferably 20.0% by weight or less, more preferably 10.0% by weight or less, still more preferably 5.0% by weight or less.

[0022] The gel particles are not particularly limited as long as they contain at least an anionic natural polymer carbohydrate, calcium ions, and water.

(Gelling Agent)

[0023] The anionic natural polymer carbohydrate is a gelling agent, whose type is not particularly limited, and is preferably a polysaccharide having a carboxyl group. For example, carrageenan, pectin, gum arabic, xanthan (xanthan gum), gellan (gellan gum), tragacanth gum, and alginic acid are preferred. Furthermore, carrageenan, pectin, gellan gum, and alginic acid are preferred because they are readily gelled in the presence of calcium ions and a carboxyl group and a cation can together create a junction zone to form a crosslinked structure. Of these, LM pectin is preferred for reasons described later. One of these substances may be used alone, or any two or more of them may be used in combination in any ratio.

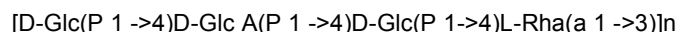
[0024] "Pectin" is a polysaccharide constituted by galacturonic acid and galacturonic acid methyl ester that are bonded through α -1,4-linkage. Pectin is known to contain several various sugars in addition to galacturonic acid. In general, pectin is classified as LM pectin with a degree of esterification of less than 50% and HM pectin with a degree of esterification

of 50% or more, and as described above, LM pectin is preferred in this embodiment.

[0025] Pectin is gelled particularly in the presence of divalent cations such as calcium ions, and a carboxyl group of galacturonic acid in pectin and a cation together create a junction zone to form a gel. Pectin having more junction zones, that is, having a lower degree of esterification has higher gelling properties.

[0026] The degree of esterification of pectin is preferably 20% or less, more preferably 12% or less, still more preferably 10% or less. The lower limit of the degree of esterification need not be set, but is typically 6% or more.

[0027] "Gellan gum" is known as a water-soluble polysaccharide synthesized by *Pseudomonas elodea*, which is a eubacterium. When a cation is added to an aqueous solution, the aqueous solution is electrically neutralized, and the water solubility of gellan gum is lowered to cause gelation. Gellan gum is a polymer compound in which repeating units composed of four sugars: two D-glucose residues, one L-rhamnose residue, and one D-glucuronic acid, are linearly linked. The repeating structure of the four sugars is as follows.



[0028] "Carrageenan" is a linear sulfur-containing polysaccharide, and is an anionic polymer compound composed of D-galactose (or 3,6-anhydro-D-galactose) and sulfuric acid.

[0029] "Alginic acid" is a polysaccharide contained mainly in brown algae. Alginic acid has a structure in which α -L-guluronic acid and β -D-mannuronic acid are bonded through 1,4-glycosidic linkage in pyranose form (CAS 9005-38-3). Alginic acid has the property of gelling upon addition of a cation.

[0030] "Gum arabic", which is also referred to as "arabic gum" or "arabic resin", is obtained by drying a secretion from a wound on the bark of *Acacia senegal* belonging to *Acacia*, *Mimosoideae*, *Fabaceae* or allied species. Gum arabic is composed mainly of a polysaccharide (polyuronic acid) and is a mixture of arabinogalactan (75-94%), arabinogalactan-protein (5-20%), and glycoprotein (1-5%). The structure of the polysaccharide has galactose in the main chain and galactose, arabinose, rhamnose, and glucuronic acid in the side chain. Gum arabic is different from hemicellulose, which constitutes cell walls, in that carboxyl groups are free, and is usually in the form of a calcium salt.

[0031] "Xanthan" is a polysaccharide and is typically produced by bacterial fermentation of corn sugar starch. Xanthan has a repeating structure including, as a unit, two glucose molecules, two mannose molecules, and a glucuronic acid molecule (CAS 11138-66-2).

[0032] "Tragacanth gum" is a polysaccharide thickener obtained by drying sap from tragacanth, which is a leguminous plant, and is a complex polysaccharide mixture of arabinose, xylose, fucose, galactose, galacturonic acid, etc. Tragacanth gum is composed mainly of polysaccharides of two types, acidic and neutral, and includes starch, cellulose, inorganic matter, etc.

[0033] The weight-average molecular weight (M_w) of the anionic natural polymer carbohydrate measured by GPC (gel permeation chromatography) and converted with a calibration curve of standard polystyrene may be, for example, 100,000 g/mol or more and 700,000 g/mol or less, or 140,000 g/mol or more and 300,000 g/mol or less, and is not limited to these ranges as long as a minimum molecular weight at which gelation is achieved is ensured. The weight-average molecular weight can be increased by adding a divalent cation and can be decreased by adding an alkali.

[0034] The content of the anionic natural polymer carbohydrate in the composition is not particularly limited, and is typically 0.01% by weight or more, preferably 0.1% by weight or more, more preferably 1.0% by weight or more, still more preferably 2.0% by weight or more, and typically 50.0% by weight or less, preferably 20% by weight or less, more preferably 10% by weight or less, still more preferably 5% by weight or less.

[0035] The content of the anionic natural polymer carbohydrate in the composition can be measured by various fractionation and separation methods and detection methods using liquid chromatography, liquid chromatography mass spectrometry, and the like.

(Gelation Aid Component)

[0036] Calcium ion serves as a gelation aid component, and examples of the source thereof (gelation aid agent) include, but are not limited to, halides (e.g., chloride), citric acid, carbonate, sulfate, phosphate, and lactate of calcium, among which calcium lactate, calcium carbonate, and calcium phosphate are preferred, and calcium lactate is particularly preferred, from the viewpoint of less influence on the flavor of the pouch product, high solubility, and pH after dissolution. One of these substances may be used alone, or any two or more of them may be used in combination in any ratio.

[0037] The calcium ion content of the composition is not particularly limited, but from the viewpoint of ensuring good fluidity and adhesion, it is typically 0.00023 mol/kg or more, preferably 0.0023 mol/kg or more, more preferably 0.023 mol/kg or more, still more preferably 0.046 mol/kg or more, and typically 1.15 mol/kg or less, preferably 0.46 mol/kg or less, more preferably 0.23 mol/kg or less, still more preferably 0.11 mol/kg or less, in terms of molar ratio relative to the weight of the composition.

[0038] Typically, the calcium ion content of the composition can be measured by atomic absorption spectrophotometry.

[0039] When a compound having a carboxyl group is used as the anionic natural polymer carbohydrate, the anionic natural polymer carbohydrate constituting the gelling agent described above is readily gelled in the presence of a divalent cation, and the carboxyl group and the cation together create a junction zone to form a gel. When there is a junction zone in the gel, the composition containing the gel has a network structure. It is desirable that the carboxyl group and the compound containing the divalent cation serving as a gelation promoting component be efficiently gelled and the carboxyl group and the divalent cation be present in a ratio of 2:1 in number. This corresponds to the case where the molar ratio of carboxyl-containing monomers in the anionic natural polymer carbohydrate to cations is 2:1. Thus, the ratio of the total number of carboxyl groups of the anionic natural polymer carbohydrate to the total number of calcium ions is preferably in the range of 100:1 to 2:1, 50:1 to 2:1, or 10:1 to 2:1.

(Other Gelation Aid Components)

[0040] The composition may contain gelation aid components other than calcium ions, and examples thereof include ions of metals such as magnesium, silver, zinc, copper, gold, and aluminum and ions of cationic polymers, which can bind the gelling agent through ionic bonds similarly to calcium ions, and examples of the source thereof (other gelation aid agents) include halides (e.g., chloride), citric acid, carbonate, sulfate, and phosphate of these metal ions and cationic polymers. One of these substances may be used alone, or any two or more of them may be used in combination in any ratio.

(Water)

[0041] The type of water contained in the composition is not particularly limited.

[0042] The water content (moisture content) of the composition is 15% by weight or more. A moisture content of less than 15% by weight leads to a rough texture and makes it difficult to produce the composition. Furthermore, from the viewpoint of ensuring good fluidity and adhesion of the composition and ease of production of the composition, the moisture content is preferably 30% by weight or more, more preferably 45% by weight or more, and typically 55% by weight or less, preferably 50% by weight or less. The moisture content can be controlled by adjusting the amount of water added or performing heat treatment or drying treatment during the production process.

[0043] The water content (moisture content) of the composition is measured using a heat-drying moisture meter (e.g., HB43-S manufactured by METIER TOLEDO). In the measurement, a sample is put into a predetermined container and heated to an attainment temperature of 100°C. The measurement is terminated when the amount of change during 60 seconds becomes 1 mg or less, and the moisture content is calculated from weighed values before and after heating.

[0044] The method of measuring the moisture content in this DESCRIPTION can be similarly applied to the measurement of the moisture content of an object other than the composition, for example, a mixture in the method for producing a composition described later.

(Nicotine)

[0045] While the composition contains nicotine, the way in which the composition contains nicotine is not particularly limited. For example, nicotine may be contained as a compound, a nicotine-containing compound such as nicotine salt or stabilized nicotine (e.g., nicotine adsorbed on ion-exchange resin) may be contained, tobacco leaves may be added as a nicotine source, or a nicotine-containing extract obtained by extracting a nicotine-containing substance such as tobacco leaves may be contained. The way in which nicotine is incorporated is also not particularly limited, and the above compound, nicotine source, or extract may be incorporated into the gel particles or may be incorporated into the composition separately from the gel particles. Among these ways, addition of a nicotine-containing compound is preferred from the viewpoint of accurate supply of nicotine and ease of handling. In general, when tobacco leaves are added, the composition and the pouch product tend to assume the color of the tobacco leaves, whereas when a colorless nicotine-containing compound is used, a white composition and a white pouch product can be provided. This is advantageous for users who prefer white pouch products.

[0046] One of the above ways may be applied alone, or two or more of the above ways may be applied in combination.

[0047] The nicotine content of the composition is not particularly limited, but from the viewpoint of user preference, it is typically 0.1% by weight or more, and typically 6.7% by weight or less. When nicotine is present as ions, the nicotine content is a content in terms of nicotine ions.

[0048] The nicotine content of the composition can be measured with a gas chromatography mass spectrometer (GC-MS).

(Other Substances)

[0049] The composition may contain substances (also referred to as "other substances") other than the above-de-

scribed anionic natural polymer carbohydrate, calcium ions, other gelation aid agents, water, and nicotine, and the other substances may be incorporated into the gel particles or may be incorporated into the composition separately from the gel particles. Examples of the other substances include a substrate, a flavor, a pH adjuster, a sweetener, a humectant, a bitterness inhibitor, a whitening agent, and an emulsifier.

[0050] The content of the other substances in the composition is not particularly limited, and their formulation can be appropriately adjusted according to the product design.

[0051] The type of the substrate is not particularly limited, and examples thereof include cellulose, microcrystalline cellulose, spherical cellulose, and porous cellulose, and from the viewpoint of flexibility in adjusting the bulk density of the composition, cellulose is preferred. One of these substances may be used alone, or any two or more of them may be used in combination in any ratio.

[0052] The content of the substrate in the composition is not particularly limited, but from the viewpoint of quality improvement by suppressing moisture leakage during production or product storage, it is typically 24% by weight or more, preferably 27% by weight or more, more preferably 30% by weight or more. There is no need to particularly limit the upper limit, but from the viewpoint of the limit of the amount of other raw materials that can be incorporated, it is typically 65% by weight or less, preferably 55% by weight or less, more preferably 50% by weight or less.

[0053] The type of the flavor is not particularly limited, and examples thereof include menthol, leaf tobacco extract, natural vegetable flavors (e.g., cinnamon, sage, herb, chamomile, kudzu, sweet Hydrangea leaves, 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, Saint John's bread, plum extract, and peach extract), saccharides (e.g., glucose, fructose, isomerized sugar, caramel, honey, and molasses), cocoas (e.g., powder and extract), esters (e.g., isoamyl acetate, linalyl acetate, isoamyl propionate, and linalyl butyrate), ketones (e.g., menthone, ionone, damascenones such as β -damascenone, and ethyl maltol), alcohols (e.g., geraniol, linalool, anethole, and eugenol), aldehydes (e.g., vanillin, benzaldehyde, and anisaldehyde), lactones (e.g., γ -undecalactone and γ -nonalactone), animal flavors (e.g., musk, ambergris, civet, and castoreum), and hydrocarbons (e.g., limonene and pinene). One of these substances may be used alone, or any two or more of them may be used in combination in any ratio.

[0054] The content of the gel particles in the composition is not particularly limited, but from the viewpoint of enjoying a desired taste and flavor, it is typically 0.1% by weight or more, preferably 1% by weight or more, more preferably 2% by weight or more, and typically 20% by weight or less, preferably 15% by weight or less, more preferably 10% by weight or less.

[0055] The type of the pH adjuster is not particularly limited, and examples thereof include sodium carbonate, sodium hydrogen carbonate, potassium carbonate, potassium hydrogen carbonate, anhydrous sodium phosphate, sodium dihydrogen phosphate, and sodium citrate. From the viewpoint of the influence on the taste of the product, sodium carbonate, potassium carbonate, and sodium dihydrogen phosphate are preferred. One of these substances may be used alone, or any two or more of them may be used in combination in any ratio.

[0056] The type of the sweetener is not particularly limited, and examples thereof include sugar alcohols such as xylitol, maltitol, and erythritol and sweeteners such as acesulfame potassium, sucralose, and aspartame. From the viewpoint of taste adjustment, sugar alcohols are preferred. One of these substances may be used alone, or any two or more of them may be used in combination in any ratio.

[0057] The bitterness inhibitor is not particularly limited, and examples thereof include soybean lecithin. Soybean lecithin is a phospholipid, and examples thereof include phosphatidylcholine, phosphatidylethanolamine, and phosphatidic acid. One of these substances may be used alone, or any two or more of them may be used in combination in any ratio.

[0058] The type of the humectant is not particularly limited, and examples thereof include glycerol and propylene glycol. From the viewpoint of product storage stability, glycerol is preferred. One of these substances may be used alone, or any two or more of them may be used in combination in any ratio.

[0059] The type of the whitening agent is not particularly limited, and examples thereof include silicon dioxide, titanium dioxide, and calcium carbonate. From the viewpoint of the influence on the taste of the product, silicon dioxide is preferred. One of these substances may be used alone, or any two or more of them may be used in combination in any ratio.

[0060] The type of the emulsifier is not particularly limited, and examples thereof include emulsifiers added to foods. The emulsifier may be, for example, at least one selected from the group consisting of sucrose fatty acid esters, organic acid glycerol fatty acid esters, polyglycerol fatty acid esters, and lecithin. Examples of sucrose fatty acid esters include sucrose palmitate and sucrose stearate. Examples of organic acid glycerol fatty acid esters include succinic acid glycerol fatty acid esters and diacetyl tartaric acid glycerol fatty acid esters. Examples of polyglycerol fatty acid esters include diglycerol fatty acid esters, triglycerol fatty acid esters, and decaglycerol fatty acid esters. One of these substances may be used alone, or any two or more of them may be used in combination in any ratio.

[0061] The content of each component described above (excluding the water content) can also be calculated from the amount of charged raw material.

(pH of Composition)

[0062] The pH of the composition at a measurement temperature of 22°C is not particularly limited, but from the viewpoint of the influence on the taste of the product, it is typically 6.0 or more, preferably 7.0 or more, more preferably 8.0 or more, and typically 10.0 or less, preferably 9.0 or less. The pH can be adjusted by controlling the amount of pH adjuster added. Not only this pH value but also pH values in this DESCRIPTION are values measured at a measurement temperature of 22°C.

[0063] The pH of the composition at a measurement temperature of 22°C is measured using a pH analyzer (e.g., LAQUA F-72 flat ISFET pH electrode manufactured by Horiba, Ltd.) by adding 20 ml of water to 2 g of the composition, shaking the mixture for 10 minutes, and measuring the supernatant.

[0064] The calibration of the instrument is performed by three-point calibration using, for example, a phthalate pH standard solution (pH: 4.01), a neutral phosphate pH standard solution (pH: 6.86), and a borate pH standard solution (pH: 9.18) (all manufactured by Wako Pure Chemical Industries, Ltd.).

(Fluidity of Composition)

[0065] As described above, in this DESCRIPTION, the fluidity of the composition is expressed in terms of shear stress at a normal stress of 5 kPa at a measurement temperature of 22°C. The normal stress of 5 kPa is employed because when a user puts the pouch product in the mouth between the inner wall of the mouth and the gums or between the inner wall of the mouth and the hypoglossal, the pressure exerted on the pouch product is typically a normal stress of 3 to 7 kPa. From the viewpoint of improving fluidity or/and adhesion, the shear stress is typically 5.99 kPa or less, preferably 5.93 kPa or less, and typically 5.02 kPa or more, preferably 5.13 kPa or more, more preferably 5.42 kPa or more. The shear stress can be increased/decreased by varying the formulation of the anionic natural polysaccharide (pectin) and Ca ions.

[0066] The shear stress of the composition at a normal stress of 5 kPa can be measured using a rheometer. For example, when an FT4 Powder Rheometer manufactured by Freeman Technology is used as a rheometer, the measurement is performed under the following measurement conditions.

- Measurement mode: standard program (25 mm_shear_9 kPa)
- Measurement temperature: 22°C
- Measurement humidity: 60%RH
- Measurement vessel: cylindrical vessel with inner diameter of 25 mm, 10 ml capacity
- Normal load: 3 to 9 kPa

[0067] Using measurement samples obtained by passing measurement raw materials each through a sieve (1.18 mm openings) to make the particles fine and uniform, the measurement is performed in accordance with the procedure of the rheometer.

(Adhesion of Composition)

[0068] As described above, in this DESCRIPTION, the adhesion of the composition is expressed in terms of shear stress at a normal stress of 0 kPa at a measurement temperature of 22°C. The normal stress of 0 kPa is a numerical value assuming a state in which no pressure is exerted other than the pressure exerted when the pouch product is pressed in the thickness direction after a user puts the pouch product in the mouth and before saliva penetrates the pouch product. From the viewpoint of improving fluidity or/and adhesion, the shear stress is typically 1.83 kPa or less, preferably 1.78 kPa or less, and typically 0.88 kPa or more, preferably 1.12 kPa or more, more preferably 1.26 kPa or more. The shear rate can be increased/decreased by varying the formulation of the anionic natural polysaccharide (pectin) and Ca ions.

[0069] As with the measurement of fluidity described above, the shear stress of the composition is measured at normal stresses of 3 kPa, 4 kPa, 5 kPa, 6 kPa, and 7 kPa, and a graph is constructed with normal stress plotted on the horizontal axis and shear stress on the vertical axis. Since shear stress changes linearly with respect to normal stress, fitting of the graph is performed, and the shear stress at a normal stress of 0 kPa is calculated from the results of the fitting. The conditions of the fitting are shown below.

[0070] From the shear stress values at the normal stresses (3 kPa, 4 kPa, 5 kPa, 6 kPa, and 7 kPa), a first-order regression line is derived by calculation. The values of the slope and the Y-intercept of the regression line are calculated. The calculated value of the Y-intercept is used as the shear stress at a normal stress of 0 kPa.

(Flow Function)

[0071] The Mohr stress circle is fitted to the first-order line related to the fitting used to calculate the shear stress at a normal stress of 0 kPa in the evaluation of adhesion described above to determine the maximum principal strength and the unconfined yield strength, and the ratio of the maximum principal strength to the unconfined yield strength (maximum principal strength/unconfined yield strength) is calculated, whereby a flow function can be evaluated.

[0072] Larger flow function (FF) values indicate higher fluidity, and the value is preferably 1.90 or more, more preferably 1.95 or more, still more preferably 2.0 or more, and typically 7.0 or less.

(Particle Size of Constituent of Dried Composition)

[0073] The size of the constituent of the composition (also referred to simply as the "composition") is not particularly limited, and, for example, the constituent of the dried composition preferably satisfies the following classification conditions.

[0074] The dried composition is preferably obtained by classification using a sieve having a sieve mesh below. From the viewpoint of ease of handling during production and control of variation in quality as well as good mouthfeel during use by users, the dried composition typically passes through a sieve having a sieve mesh of 15 mm (≤ 15 mm), preferably passes through a sieve having a sieve mesh of 10 mm (≤ 10 mm), more preferably passes through a sieve having a sieve mesh of 5 mm (≤ 5 mm), still more preferably passes through a sieve having a sieve mesh of 3.2 mm (≤ 3.2 mm). For example, when all of the dried gel particles pass through a sieve having a sieve mesh of X mm, it indicates that the gel particles have a maximum particle size of X mm or less when dried.

[0075] The dried composition can be obtained by holding the composition at 70°C to 80°C for about 3 hours to dryness.

[0076] The maximum particle size of the composition can be appropriately increased/decreased by varying the formulation of the anionic natural polysaccharide (pectin) and Ca ions or the amount of water contained.

[0077] From the viewpoint of improving fluidity or/and adhesion, there is no need to set the lower limit the particle size of the dried composition, but from the viewpoint of preventing leakage from the pouch, the maximum particle size of the dried composition is typically 0.3 μ m or more.

[Pouch]

[0078] The pouch (packaging material) may be any known pouch that can package the composition, that is insoluble in water, and that is permeable to liquids (e.g., water and saliva) and water-soluble components in the composition. The material of the pouch may be, for example, a cellulose-based nonwoven fabric, and a commercially available nonwoven fabric may be used. The pouch product can be produced by forming a sheet made of such a material into a bag shape, putting the composition into the bag, and sealing the bag by means of heat sealing or other means.

[0079] The basis weight of the sheet is not particularly limited, and is typically 12 gsm or more and 54 gsm or less, preferably 24 gsm or more and 30 gsm or less.

[0080] The thickness of the sheet is not particularly limited, and is typically 100 μ m or more and 300 μ m or less, preferably 175 μ m or more and 215 μ m or less.

[0081] At least one of the inner surface and the outer surface of the pouch may be partially coated with a water-repellent material. As the water-repellent material, a water-repellent fluorocarbon resin is suitable. Specific examples of such a water-repellent fluorocarbon resin include AsahiGuard (registered trademark) manufactured by AGC Inc. The water-repellent fluorocarbon resin is applied, for example, to packing materials for foods and products containing fats and oils, such as confectioneries, dairy products, daily dishes, fast foods, and pet foods. Therefore, such a water-repellent fluorocarbon resin is safe even when applied to a pouch to be placed in the oral cavity. The water-repellent material need not be a fluorocarbon resin and may be any material having a water-repellent effect, such as a paraffin resin, a silicon resin, or an epoxy resin.

[0082] The pouch may contain optional components, examples of which include materials for controlling flavor and taste, flavors, additives, tobacco extracts, and pigments. These components may be incorporated in any manner. For example, the components may be applied to or infiltrated into the surface of the pouch, or when the pouch is made of fibers, the components may be incorporated into the fibers.

[0083] Furthermore, the appearance of the pouch is not particularly limited and may not only be non-transparent but also be translucent or transparent, and in this case, the composition packaged in the pouch can be seen through.

[Pouch Product]

[0084] The pouch product is not particularly limited as long as it includes the composition and the pouch for packaging the composition (the composition enclosed in the pouch).

[0085] The size and weight of the pouch product are not particularly limited. For the size of the pouch product before use, the long side may be 25 mm or more (e.g., 28 mm, 35 mm, or 38 mm) and 40 mm or less, or 28 mm or more and 38 mm or less, and the short side may be 10 mm or more and 20 mm or less, or 14 mm or more and 18 mm or less. The weight of the pouch product before use may be 0.1 g or more and 2.0 g or less, or 0.3 g or more and 1.0 g or less.

[0086] The ratio of the weight of the composition to the total weight of the pouch product is not particularly limited, and is typically 80% by weight or more, preferably 85% by weight or more, more preferably 90% by weight or more, and typically 99% by weight or less, preferably 97% by weight or less, more preferably 95% by weight or less.

[0087] In the measurement of the properties in this DESCRIPTION, a measurement sample is held for 48 hours or more in an environment similar to the measurement environment before the measurement. Unless otherwise specified, the measurement temperature is normal temperature ($22 \pm 2^\circ\text{C}$), the measurement humidity is normal humidity ($60 \pm 5\%\text{RH}$), and the measurement pressure is normal pressure (atmospheric pressure).

<Method for Producing Nicotine Supply Oral Pouch Product>

[0088] A method for producing a nicotine supply oral pouch product according to another embodiment of the present invention (also referred to simply as a "method for producing a nicotine supply oral pouch product" or a "production method") includes a composition production step of producing a composition containing gel particles containing at least an anionic natural polymer carbohydrate, a calcium ion supplier, and water.

[0089] The composition contains nicotine.

[0090] The water content of the composition is 15% by weight or more.

[Composition Production Step]

[0091] The composition production step is not particularly limited as long as the composition can be produced. An example of a method for producing the composition is shown below. As raw materials shown below, the raw materials described above can be used.

[0092] First, an anionic natural polymer carbohydrate such as pectin, a nicotine source such as a stabilized nicotine compound, a substrate such as cellulose, and a whitening agent such as silicon dioxide are mixed to obtain a mixture (mixture before calcium source addition) (step of preparation of mixture before calcium source addition). Thereafter, an aqueous solution containing a calcium source such as calcium lactate and an aqueous solution containing a pH adjuster such as anhydrous sodium phosphate are added to the mixture (e.g., added by spraying), and the resultant is mixed to obtain a mixture (mixture before heating) (step of preparation of mixture before heating).

[0093] The calcium source may be supplied in the form of an aqueous solution or a solid, but when it is supplied in the form of a liquid such as an aqueous solution, calcium can be mixed with other raw materials in the form of calcium ions to achieve higher efficiency of contact with the anionic natural polymer carbohydrate than when it is supplied as a solid; thus, the calcium source is preferably supplied in the form of an aqueous solution. The calcium source may be added after the mixture containing the anionic natural polymer carbohydrate is prepared as described above or may be mixed first together with the anionic natural polymer carbohydrate, but from the viewpoint of forming fine and uniform gel particles, the calcium source is preferably added after the mixture containing the anionic natural polymer carbohydrate is prepared. On the other hand, from the viewpoint of handling, it is not preferable to use the anionic natural polymer carbohydrate such as pectin in the form of an aqueous solution because the viscosity increases when the anionic natural polymer carbohydrate is formed into an aqueous solution.

[0094] In the addition of the aqueous solutions after the step of preparation of mixture before calcium source addition, water may be added together with each aqueous solution in order to achieve a desired water content.

[0095] From the viewpoint of sufficiently melting the anionic natural polymer carbohydrate in advance, a sufficient amount of water is preferably taken into the mixture before the following heating step (gel particle formation step). For example, at a point in time after completion of the step of preparation of mixture before heating, the moisture content of the mixture is preferably 10% by weight or more and 50% by weight or less, more preferably 20% by weight or more and 40% by weight or less.

[0096] The mixture before heating is preferably subjected to pH adjustment. For example, a pH adjuster such as sodium dihydrogen phosphate is preferably added with its amount adjusted such that the mixture before heating becomes acidic. Specifically, the pH of the mixture before heating is preferably 3.0 or more and 6.0 or less, more preferably 4.0 or more and 6.0 or less.

[0097] By adjusting the pH to 6.0 or less in this step, the process can proceed to the heating step while maintaining the nicotine contained in the mixture before heating in a stable state.

[0098] The mixture before heating is heated to hydrate the anionic natural polymer carbohydrate and promote contact between the anionic natural polymer carbohydrate and calcium ions, whereby gel particles are formed to obtain a mixture containing the gel particles (gel particle-containing mixture) (heating step).

[0099] The method of heating in the heating step is not particularly limited. For example, increase of the jacket temperature of a mixer holding the mixture (jacket heating), injection of steam into the mixture, or both may be used. The temperature of the mixture during heating is not particularly limited, but from the viewpoint of efficiently hydrating the anionic natural polymer carbohydrate, it is preferably 60°C or higher and 90°C or lower, more preferably 70°C or higher and 80°C or lower. The heating time is not particularly limited, but from the viewpoint of efficiently reacting the anionic natural polymer carbohydrate with Ca ions and from the viewpoint of appropriately adjusting the moisture in the composition, it is preferably 1 hour or more and 3 hours or less, more preferably 1 hour or more and 2 hours or less.

[0100] After the heating step, the mixture may be dried (drying step). Thereafter, cooling may be performed. The cooling may be natural cooling or may be performed by using some cooling means (cooling step). By performing drying, for example, the moisture content of the mixture can be adjusted to 5% to 45% by weight. This makes it easy to adjust the moisture content of the composition as a target.

[0101] To the gel particle-containing mixture obtained in the heating step (or the drying step or the cooling step), an aqueous solution containing a pH adjuster such as potassium carbonate is further added.

[0102] The pH adjuster such as potassium carbonate is preferably added with its amount adjusted such that the mixture after heating becomes alkaline. Specifically, the pH of the mixture before heating is preferably 6.0 or more and 10.0 or less, more preferably 8.0 or more and 9.0 or less.

[0103] A sweetener such as acesulfame potassium, a flavor such as menthol, a bitterness inhibitor such as soybean lecithin, and a humectant such as glycerol are added (step of additive addition to gel particle-containing mixture) to obtain a desired composition.

[0104] When these additives are added, they may be added in the form of solids or aqueous solutions in which they are dissolved. In the case of addition in the form of an aqueous solution, the additive may be dissolved in a predetermined amount of water in advance so as to achieve the final moisture content of the pouch product and added.

[Packaging Step]

[0105] The composition obtained in the composition production step is packaged with a packaging material to obtain a pouch product (packaging step). The method of packaging is not particularly limited, and a known method can be applied. For example, a known method such as putting the composition into a bag-shaped nonwoven fabric and then sealing the nonwoven fabric can be used.

[0106] After the composition is put into the packaging material and the packaging material is then sealed in the packaging step, water may further be added in order to provide the composition with a desired water content (water addition step). For example, when the water content of the target composition is 50% by weight and the water content of the composition obtained in the composition production step is 15% by weight, the difference, i.e., 35% by weight of water is added.

<Applications of Pouch Product>

[0107] Examples of applications (modes of use) of the pouch product include, but are not limited to, oral tobaccos such as chewing tobacco, snuff, and compressed tobacco and nicotine-containing preparations called nicotine pouches. These are inserted between the lips and gums in the oral cavity to enjoy their taste and flavor.

EXAMPLES

[0108] The present invention will now be described more specifically with reference to Examples. However, the present invention should not be construed as being limited to the following Examples.

<Experiment 1>

[Preparation of Composition]

(Example 1)

[0109] After 75.0 g of pectin (Classic CU902 manufactured by H&F), 680.0 g of nicotine polacrilex (Nicotine Polacrilex 20% manufactured by Contraf nicotex), 2175 g of cellulose (VITACEL L600-30 manufactured by J. RETTENMAIER & SOHNE), and 75.0 g of silicon dioxide (silica) (SYLOPAGE 720 manufactured by Fuji Silysia Chemical Ltd.) were mixed, 35.3 g of an aqueous calcium lactate solution (calcium lactate (granules) manufactured by Taihei Chemical Industrial Co., Ltd.) and 302.1 g of an aqueous anhydrous sodium phosphate solution (Monosodium phosphate anhydrous, FG (MSP A FG) manufactured by Univar B.V) were added to the mixture, and these were mixed until homogeneous to

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obtain a mixture A (moisture content: 23.5% by weight) having a pH of 6.2.

[0110] The mixture A obtained was subjected to jacket heating (can wall temperature: 100°C) for 1 hour so that the temperature of the raw materials became 70°C to 80°C (moisture content after heating: 6.2% by weight, pH after heating: 6.0 (measured by the same measurement method as in the case of the tobacco filler described above)). The mixture

was then cooled for 1 hour at an ambient temperature of 20°C to obtain a mixture B (pH after cooling: 6.2).
[0111] To the mixture B obtained by cooling, 256.1 g of an aqueous potassium carbonate solution (POTASSIUM CARBONATE manufactured by Univar B.V) was added to obtain a mixture C.

[0112] Lastly, to adjust the moisture content, 0 g of water was added to the mixture C (i.e., no additional water was added) to obtain 200 g of a composition 1 of Example 1 (moisture content: 15.10% by weight, pH: 7.8).

(Example 2)

[0113] A composition 2 of Example 2 (moisture content: 18.47% by weight) in an amount of 100 g was prepared in the same manner as the composition 1 except that the amount of water added to the mixture C was changed from 0 g to 1.71 g.

(Example 3)

[0114] A composition 3 of Example 3 (moisture content: 32.06% by weight) in an amount of 200 g was prepared in the same manner as the composition 1 except that the amount of water added to the mixture C was changed from 0 g to 42.57 g.

(Example 4)

[0115] A composition 4 of Example 4 (moisture content: 46.98% by weight) in an amount of 200 g was prepared in the same manner as the composition 1 except that the amount of water added to the mixture C was changed from 0 g to 108.73 g.

(Example 5)

[0116] A composition 5 of Example 5 (moisture content: 52.49% by weight) in an amount of 100 g was prepared in the same manner as the composition 1 except that the amount of water added to the mixture C was changed from 0 g to 66.80 g.

(Comparative Example 1)

[0117] A composition 6 of Comparative Example 1 (moisture content: 18.18% by weight, pH: 7.8) in an amount of 100 g was prepared in the same manner as the composition 1 except that pectin and the aqueous calcium lactate solution were not added and the amount of water added to the mixture C was changed from 0 g to 3.46 g.

(Comparative Example 2)

[0118] A composition 7 of Comparative Example 2 (moisture content: 31.46% by weight) in an amount of 200 g was prepared in the same manner as the composition 1 except that pectin and the aqueous calcium lactate solution were not added and the amount of water added to the mixture C was changed from 0 g to 43.00 g.

(Comparative Example 3)

[0119] A composition 8 of Comparative Example 3 (moisture content: 46.60% by weight) in an amount of 200 g was prepared in the same manner as the composition 1 except that pectin and the aqueous calcium lactate solution were not added and the amount of water added to the mixture C was changed from 0 g to 109.27 g.

[Property Evaluation]

(Moisture Content)

[0120] The water content (moisture content) of each composition was measured using a heat-drying moisture meter (e.g., HB 43-S manufactured by METIER TOLEDO).

- Measurement mode: FAST
- Attainment temperature (stable): 100°C
- Measurement end conditions: AUTO60 (the amount of change during 60 seconds: 1 mg or less)
- Result expression: Moisture%

(pH)

[0121] The pH of the composition at a measurement temperature of 22°C was measured using a pH analyzer (e.g., LAQUA F-72 flat ISFET pH electrode manufactured by Horiba, Ltd.).

[0122] The calibration of the instrument was performed by three-point calibration using, for example, a phthalate pH standard solution (pH: 4.01), a neutral phosphate pH standard solution (pH: 6.86), and a borate pH standard solution (pH: 9.18) (all manufactured by Wako Pure Chemical Industries, Ltd.).

(Fluidity)

[0123] The shear stress of the composition at a normal stress of 5 kPa was measured under the following measurement conditions using an FT4 Powder Rheometer manufactured by Freeman Technology as a rheometer, and the shear stress at a normal stress of 5 kPa was employed.

- Measurement mode: standard program (25 mm_shear_9 kPa)
- Measurement temperature: 22°C
- Measurement humidity: 60%RH
- Measurement vessel: cylindrical vessel with inner diameter of 25 mm, 10 ml capacity
- Normal stress: 3 to 9 kPa

[0124] Using measurement samples obtained by passing measurement raw materials each through a sieve (1.18 mm openings) to make the particles fine and uniform, the measurement was performed in accordance with the procedure of the rheometer.

(Adhesion)

[0125] As with the measurement of fluidity described above, the shear stress of the composition was measured at normal stresses of 3 kPa, 4 kPa, 5 kPa, 6 kPa, and 7 kPa, and a graph was constructed with normal stress plotted on the horizontal axis and shear stress on the vertical axis. Since shear stress changes linearly with respect to normal stress, fitting of the graph was performed, and the shear stress at a normal stress of 0 kPa was calculated from the results of the fitting. The conditions of the fitting are shown below.

[0126] From the shear stress values at the normal stresses (3 kPa, 4 kPa, 5 kPa, 6 kPa, and 7 kPa), a first-order regression line is derived by calculation. The values of the slope and the Y-intercept of the regression line are calculated. The calculated value of the Y-intercept is used as the shear stress at a normal stress of 0 kPa.

[0127] The amounts of raw materials of the above compositions and evaluation results of the properties are listed in Table 1 below. The raw materials shown in Table 1 are not all raw materials contained but some of the raw materials contained. Numerical values for the raw materials shown in the table are not contents of the components in the compositions but amounts charged. In Table 1, "≤X" in the row of Maximum particle size of composition means that the maximum particle size of the dried composition is X or less.

[0128] The symbol "-" in Table 1 indicates no addition.

[0129] The measurement results of the shear stress of the compositions at normal stresses of 3 kPa, 4 kPa, 5 kPa, 6 kPa, and 7 kPa described in the section of Adhesion above are shown in Table 2, and graphs obtained by plotting the measurement results of Example 2 and Comparative Example 1 (moisture content: about 18% by weight), Example 3 and Comparative Example 2 (moisture content: about 30% by weight), and Example 4 and Comparative Example 3 (moisture content: about 45% by weight) are shown in Figs. 1 to 3.

[Table 1]

		Example 1	Example 2	Example 3	Example 4	Example 5	Comparative Example 1	Comparative Example 2	Comparative Example 3
		g	g	g	g	g			
Raw materials	Nicotine polacrilex	680.0	680.0	680.0	680.0	680.0	680.0	680.0	680.0
	Cellulose	2175.0	2175.0	2175.0	2175.0	2175.0	2175.0	2175.0	2175.0
	Pectin	75.0	75.0	75.0	75.0	75.0	-	-	-
	Silica	75.0	75.0	75.0	75.0	75.0	75.0	75.0	75.0
	Aqueous Ca lactate solution	35.3	35.3	35.3	35.3	35.3	-	-	-
	Aqueous anhydrous Na phosphate solution	302.1	302.1	302.1	302.1	302.1	302.1	302.1	302.1
	Aqueous K carbonate solution	256.1	256.1	256.1	256.1	256.1	256.1	256.1	256.1
Properties	Moisture content	15.53	18.47	32.06	46.60	52.49	18.18	31.46	46.60
	pH of composition	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8
	Shear stress (normal stress: 5 kPa)	5.02	5.13	5.42	5.93	5.99	5.45	5.62	6.29
	Shear stress (normal stress: 0 kPa)	0.88	1.12	1.26	1.76	1.83	1.33	1.36	2.03
	Maximum particle size of composition	≤3.2	≤3.2	≤3.2	≤3.2	≤3.2	≤3.2	≤3.2	≤3.2

[Table 2]

Normal stress	Shear stress (kPa)							
	Example 1	Example 2	Example 3	Example 4	Example 5	Comparative Example 1	Comparative Example 2	Comparative Example 3
3 kPa	3.37	3.46	3.64	4.17	4.18	3.74	3.83	4.48
4 kPa	4.15	4.32	4.58	5.13	5.11	4.64	4.77	5.43
5 kPa	5.02	5.13	5.42	5.93	5.99	5.45	5.62	6.29
6 kPa	5.83	5.84	6.17	6.77	6.67	6.22	6.43	7.15
7 kPa	6.65	6.65	6.94	7.5	7.47	7.03	7.21	7.81

[0130] From Table 1 above, it has been found that the compositions of Examples 1 to 5 satisfying the requirements of the above embodiment each have a lower shear stress at a normal stress of 5 kPa and a lower shear stress at a normal stress of 0 kPa, that is, higher fluidity and higher adhesion, than the compositions of Comparative Examples 1 to 3 not satisfying the requirements. Specifically, it has been found that the fluidity and adhesion are improved by using pectin and the aqueous calcium lactate solution in combination.

<Experiment 2>

[Preparation of Composition]

(Examples 1 to 5)

[0131] As compositions of Examples, the compositions 1 to 5 of Examples 1 to 5 above were used.

(Comparative Example 4)

[0132] A composition 9 of Comparative Example 4 (moisture content: 16.12% by weight, pH: 7.8) in an amount of 200 g was prepared in the same manner as the composition 1 except that the aqueous calcium lactate solution was not added.

(Comparative Example 5)

[0133] A composition 10 of Comparative Example 5 (moisture content: 47.06% by weight) in an amount of 200 g was prepared in the same manner as the composition 1 except that the aqueous calcium lactate solution was not added and the amount of water added to the mixture C was changed from 0 g to 105.33 g.

(Comparative Example 6)

[0134] A composition 11 of Comparative Example 6 (moisture content: 55.97% by weight) in an amount of 100 g was prepared in the same manner as the composition 1 except that the aqueous calcium lactate solution was not added and the amount of water added to the mixture C was changed from 0 g to 86.09 g.

[Property Evaluation]

[0135] These compositions were each subjected to the same property evaluations as in Experiment 1 above. The evaluation results of the properties are shown in Table 3 below. The raw materials shown in Table 3 are not all raw materials contained but some of the raw materials contained. Numerical values for the raw materials shown in the table are not contents of the components in the compositions but amounts charged. In Table 3, " \leq X" in the row of Maximum particle size of composition means that the maximum particle size of the dried composition is X or less.

[0136] The measurement results of the shear stress of the compositions at normal stresses of 3 kPa, 4 kPa, 5 kPa, 6 kPa, and 7 kPa described in the section of Adhesion above are shown in Table 4, and graphs obtained by plotting the measurement results of Example 1 and Comparative Example 4 (moisture content: about 15% by weight), Example 4 and Comparative Example 5 (moisture content: about 45% by weight), and Example 5 and Comparative Example 6 (moisture content: about 50% by weight) are shown in Figs. 4 to 6.

[Table 3]

		Example 1	Example 2	Example 3	Example 4	Example 5	Comparative Example 4	Comparative Example 5	Comparative Example 6
		g	g	g	g	g	g	g	g
Raw materials	Nicotine polacrilex	680.0	680.0	680.0	680.0	680.0	680.0	680.0	680.0
	Cellulose	2175.0	2175.0	2175.0	2175.0	2175.0	2175.0	2175.0	2175.0
	Pectin	75.0	75.0	75.0	75.0	75.0	75.0	75.0	75.0
	Silica	75.0	75.0	75.0	75.0	75.0	75.0	75.0	75.0
	Aqueous Ca lactate solution	35.3	35.3	35.3	35.3	35.3	-	-	-
	Aqueous anhydrous Na phosphate solution	302.1	302.1	302.1	302.1	302.1	302.1	302.1	302.1
	Aqueous K carbonate solution	256.1	256.1	256.1	256.1	256.1	256.1	256.1	256.1
Properties	Moisture content	15.53	18.47	32.06	46.60	52.49	16.12	47.06	55.97
	pH of composition	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8
	Shear stress (normal stress: 5 kPa)	5.02	5.13	5.42	5.93	5.99	5.35	6.17	6.17
	Shear stress (normal stress: 0 kPa)	0.88	1.12	1.26	1.76	1.83	1.22	2.00	1.89
	Maximum particle size of composition	≤3.2	≤3.2	≤3.2	≤3.2	≤3.2	≤3.2	≤3.2	≤3.2

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[Table 4]

Normal stress	Shear stress (kPa)					
	Example 1	Example 2	Example 3	Example 4	Example 5	Comparative Example 6
3 kPa	3.37	3.46	3.64	4.17	4.18	4.37
4 kPa	4.15	4.32	4.58	5.13	5.11	5.25
5 kPa	5.02	5.13	5.42	5.93	5.99	6.17
6 kPa	5.83	5.84	6.17	6.77	6.67	7
7 kPa	6.65	6.65	6.94	7.5	7.47	7.74

[0137] From Table 3 above, it has been found that the compositions of Examples 1 to 5 satisfying the requirements of the above embodiment each have a lower shear stress at a normal stress of 5 kPa and a lower shear stress at a normal stress of 0 kPa, that is, higher fluidity and higher adhesion, than the compositions of Comparative Examples 4 to 6 not satisfying the requirements. Specifically, it has been found that the fluidity and adhesion are improved by using the aqueous calcium lactate solution.

[0138] Furthermore, taken together with Table 1 for Experiment 1 above, it can be seen that comparison of the compositions of Example 4, Comparative Example 2, and Comparative Example 5 each having a moisture content of about 45% by weight shows that the shear stress at a normal stress of 5 kPa and the shear stress at a normal stress of 0 kPa increase in the order mentioned. That is, it has been found that the fluidity and adhesion are improved by adding pectin, and, furthermore, the fluidity and adhesion are further improved by adding the aqueous calcium lactate solution.

<Experiment 3>

[Preparation of Composition]

(Comparative Examples 4 to 6)

[0139] In this experiment, Comparative Examples 4 to 6 above were used.

(Comparative Example 7)

[0140] A composition 12 of Comparative Example 7 (pH: 8.6, moisture content: 7.0% by weight) in an amount of 100 g was obtained by mixing 7.7 g of pectin (Classic CU902 manufactured by H&F), 2.2 g of nicotine polacrilex (Nicotine Polacrilex 20% manufactured by Contraf nicotex), 32.8 g of microcrystalline cellulose (HICEL 90M MCC manufactured by Brenntag Nordic), 6.3 g of sodium carbonate (SODIUM CARBONATE ANHYDROUS manufactured by UNIVAR), and 41.2 g of maltitol (C*Maltidex CH 16385 manufactured by CALDIC NORDIC) until homogeneous.

[0141] In Comparative Example 7, as can be seen from the above production method, addition of aqueous solutions or water and heat treatment were not performed. The water shown by the above moisture content is derived from the water originally contained in the above raw materials.

[Property Evaluation]

(Sensory Evaluation)

[0142] The compositions of Comparative Examples 4 to 7 were each put into a nonwoven fabric (manufactured by BFF technical fabrics, basis weight: 27.0 g/m²) so as to be 0.65 g per nonwoven fabric, and then hermetically sealed by heat sealing to produce pouch products, and sensory evaluation was performed. The sensory evaluation was performed in the following manner. Five expert panelists for sensory evaluation of pouch products put each of the pouches produced using the above compositions in their mouths, and evaluated various items such as mouthfeel, flavor, and satisfaction on a scale from good to poor.

[0143] As a result of the sensory evaluation, in Comparative Example 7 in which water was not added, as compared with Comparative Examples 4, 5, and 6 in which water was added (including addition of water in the form of an aqueous solution), there was a tendency to have a feeling of a foreign body, a feeling of dry mouth, and poor affinity for saliva in the oral cavity when the pouch was put in the mouth

<Experiment 4>

[Preparation of Composition]

(Example 6)

[0144] A mixture D was obtained by mixing 885 g of cellulose (VITACEL L600-30 manufactured by J. RETTENMAIER & SOHNE), 120.0 g of nicotine polacrilex (Nicotine Polacrilex 20% manufactured by Contraf nicotex), 0.6 g of tobacco powder, 339 g of sodium dihydrogen phosphate (OMNISAL manufactured by Fosfa), 282.0 g of sodium carbonate (SODIUM CARBONATE ANHYDROUS manufactured by UNIVAR), 87.0 g of glycerol fatty acid ester (POEM DO-100V manufactured by Riken Vitamin Co., Ltd.), 171 g of sodium chloride (Suprasel Classic manufactured by Nouryon), 45 g of calcium lactate (calcium lactate (granules) manufactured by Taihei Chemical Industrial Co., Ltd.), and 66 g a sweetener until homogeneous. Thereafter, 84 g of a flavor was added to the mixture D obtained, and 7.5 g of gellan gum (KELCOGEL

manufactured by San-Ei Gen F.F.L, Inc.) and 909 g of water were further added and mixed to obtain a composition 13 (moisture content: 30.3% by weight).

(Comparative Example 8)

[0145] A mixture D was prepared in the same manner as in Example 6 except that the amount of cellulose was changed from 885 g to 888 g, and the amount of sodium dihydrogen phosphate from 339 g to 342 g. Thereafter, 84 g of a flavor was added to the mixture D obtained, and 912 g of water was further added and mixed to obtain a composition 13 (moisture content: 30.3% by weight).

[0146] The formulation of the raw materials in the above examples is shown in Table 5.

[Table 5]

	Example6	Comparative Example 8
	Weight (g)	Weight (g)
Cellulose	885	888
Nicotine polacrilex	120	120
Tobacco powder	0.6	0.6
Sodium dihydrogen phosphate	339	342
Sodium carbonate	282	282
Fatty acid ester	87	87
Sodium chloride	171	171
Calcium lactate	45	45
Sweetener	66	66
Gellan gum	7.5	-
Flavor	84	84
Water	909	909
Total	2996.1	2997.6

[Property Evaluation]

(Fluidity and Adhesion)

[0147] The above compositions were each evaluated for fluidity and adhesion in the same manner as in Experiment 1 except that the inner diameter of the measurement vessel was changed from 25 mm to 50 mm, and the capacity of the measurement vessel from 10 ml to 85 ml. The evaluation results are shown in Table 6 below and Fig. 7. Here, shear stresses of the compositions at normal stresses of 3 kPa, 4 kPa, 5 kPa, and 6 kPa were evaluated.

[Table 6]

Normal stress	Shear stress (kPa)	
	Example6	Comparative Example 8
0 kPa	2.30	3.48
3 kPa	5.06	5.54
4 kPa	5.91	6.33
5 kPa	6.94	7.10
6 kPa	7.53	7.97

[0148] From Table 6 above and Fig. 7, it has been found that the compositions of Example 6 satisfying the requirements

of the above embodiment has a lower shear stress at a normal stress of 5 kPa and a lower shear stress at a normal stress of 0 kPa, that is, higher fluidity and higher adhesion, than the compositions of Comparative Example 8 not satisfying the requirements. Specifically, it has been found that the fluidity and adhesion are improved by the composition using gellan gum and the aqueous calcium lactate solution in combination.

[0149] It has also been found that the shear stresses at lower normal stresses are lower in Examples 6 and 7 than in Comparative Example 8. This is probably due to reduction in adhesion between particles at low normal stresses.

[Property Evaluation]

(Flow Function)

[0150] The Mohr stress circle was fitted to the first-order line related to the fitting used to calculate the shear stress at a normal stress of 0 kPa in the evaluation of adhesion described above to determine the maximum principal strength and the uniaxial collapse strength, and the ratio of the maximum principal strength to the unconfined yield strength (maximum principal strength/unconfined yield strength) was calculated and evaluated as a flow function. The evaluation results are shown in Table 7.

[Table 7]

Normal stress	Flow Function	
	Example 6	Comparative Example 8
0 kPa	2.21	1.82

[0151] From Table 7 above, it has been found that the composition of Example 6 satisfying the requirements of the above embodiment has higher fluidity than the composition of Comparative Example 8 not satisfying the requirements.

[0152] As described above, the present invention can provide a nicotine supply oral pouch product having improved fluidity or improved adhesion and a method for producing the nicotine supply oral pouch product.

Claims

1. A nicotine supply oral pouch product comprising:

a composition containing gel particles; and
a pouch for packaging the composition,
wherein the gel particles contain at least an anionic natural polymer carbohydrate, calcium ions, and water,
the composition contains nicotine, and
a water content of the composition is 15% by weight or more.

2. The nicotine supply oral pouch product according to claim 1, wherein the anionic natural polymer carbohydrate has a carboxyl group.

3. The nicotine supply oral pouch product according to claim 2, wherein the anionic natural polymer carbohydrate is LM pectin.

4. The nicotine supply oral pouch product according to claim 3 or 4, wherein in the composition, a ratio of a total number of carboxyl groups of the anionic natural polymer carbohydrate to a total number of the calcium ions is 100: 1 to 2: 1.

5. The nicotine supply oral pouch product according to any one of claims 1 to 4, wherein a constituent of the composition has a maximum particle size of 15 mm or less when dried.

6. The nicotine supply oral pouch product according to any one of claims 1 to 5, wherein the pouch is a nonwoven fabric.

7. A method for producing a nicotine supply oral pouch product, comprising a composition production step of producing a composition containing gel particles comprising at least an anionic natural polymer carbohydrate, a calcium ion supplier, and water,

wherein the composition comprises nicotine, and
a water content of the composition is 15% by weight or more.

- 5 **8.** The method for producing a nicotine supply oral pouch product according to claim 7, wherein the calcium ion supplier is a liquid.
9. The method for producing a nicotine supply oral pouch product according to claim 7 or 8, wherein the calcium ion supplier comprises calcium lactate.

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FIG. 1

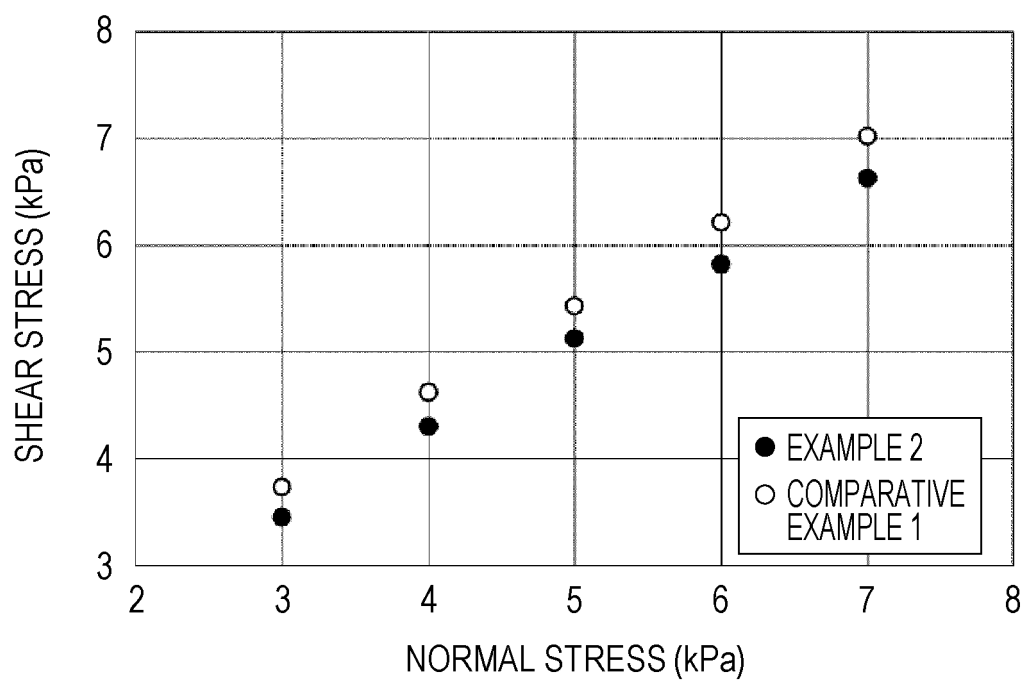


FIG. 2

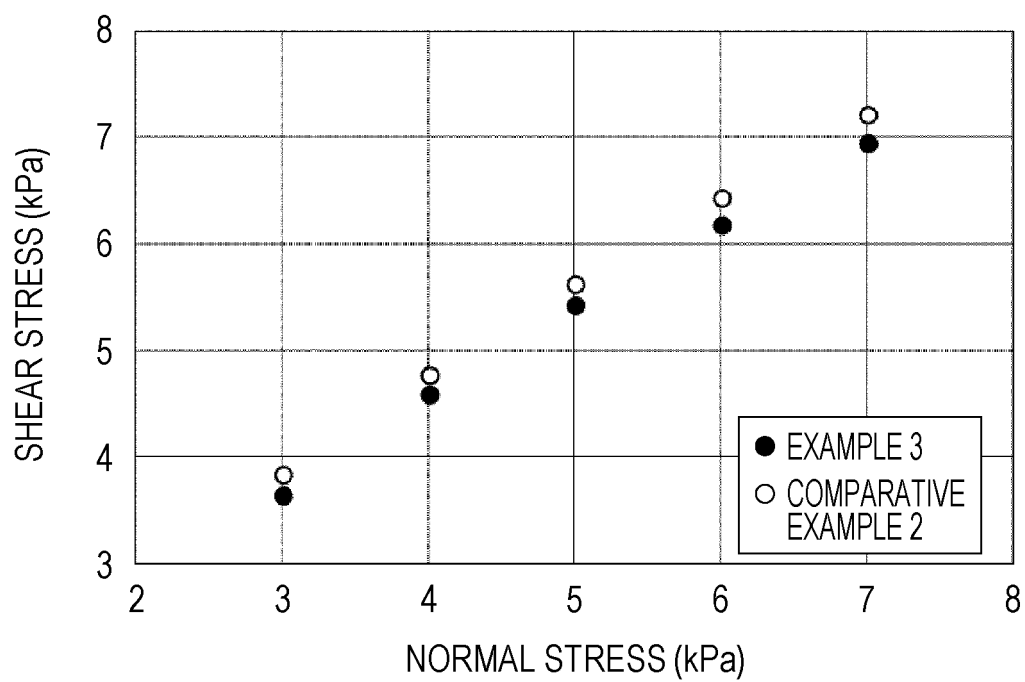


FIG. 3

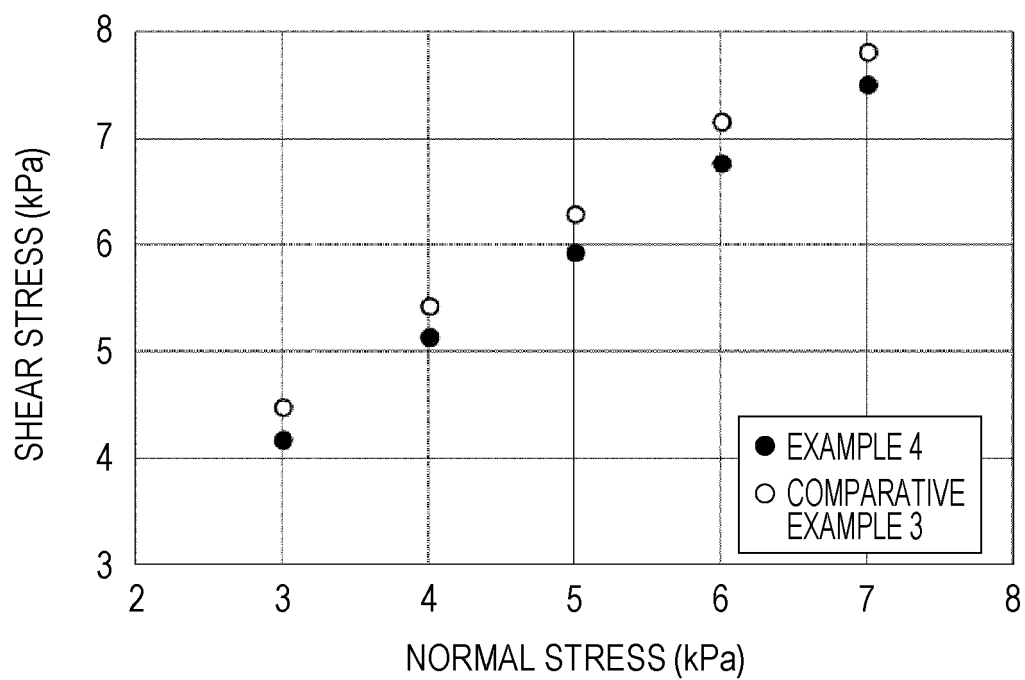


FIG. 4

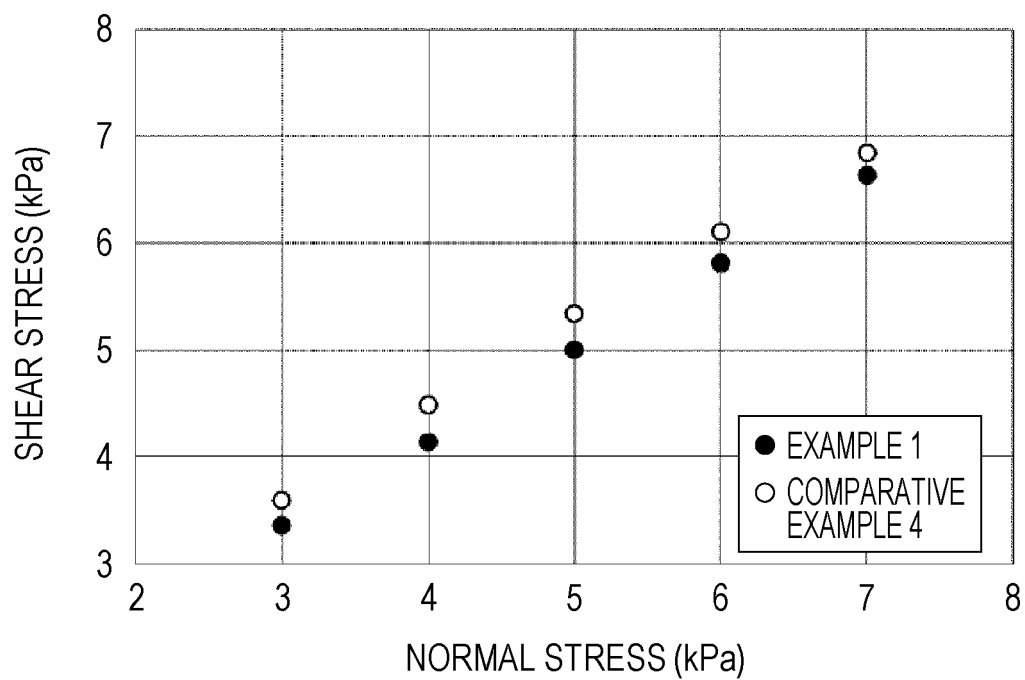


FIG. 5

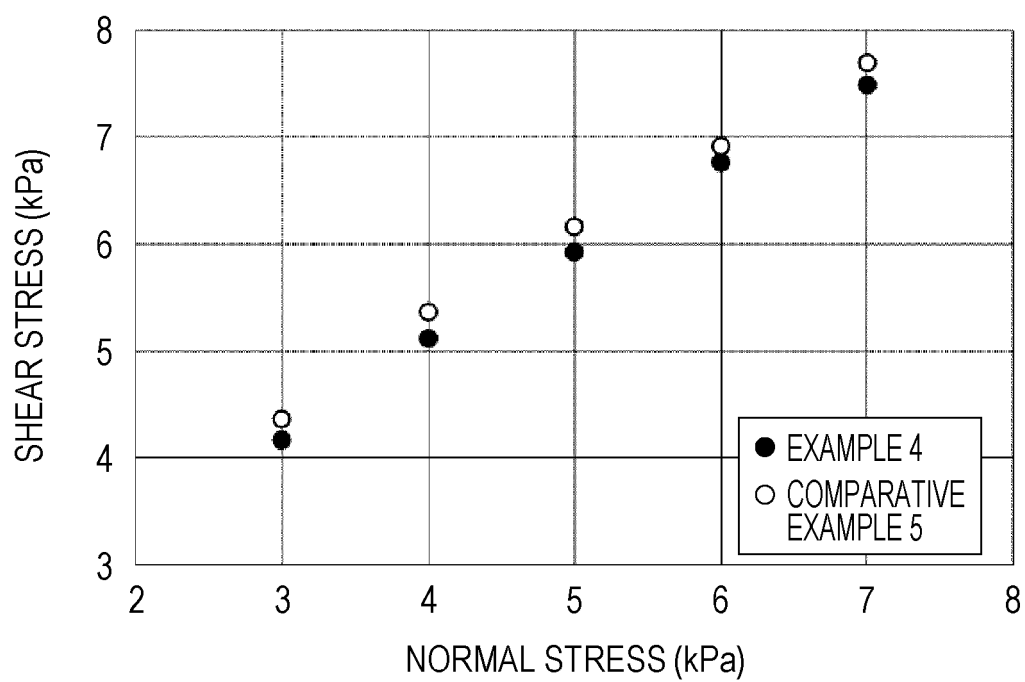


FIG. 6

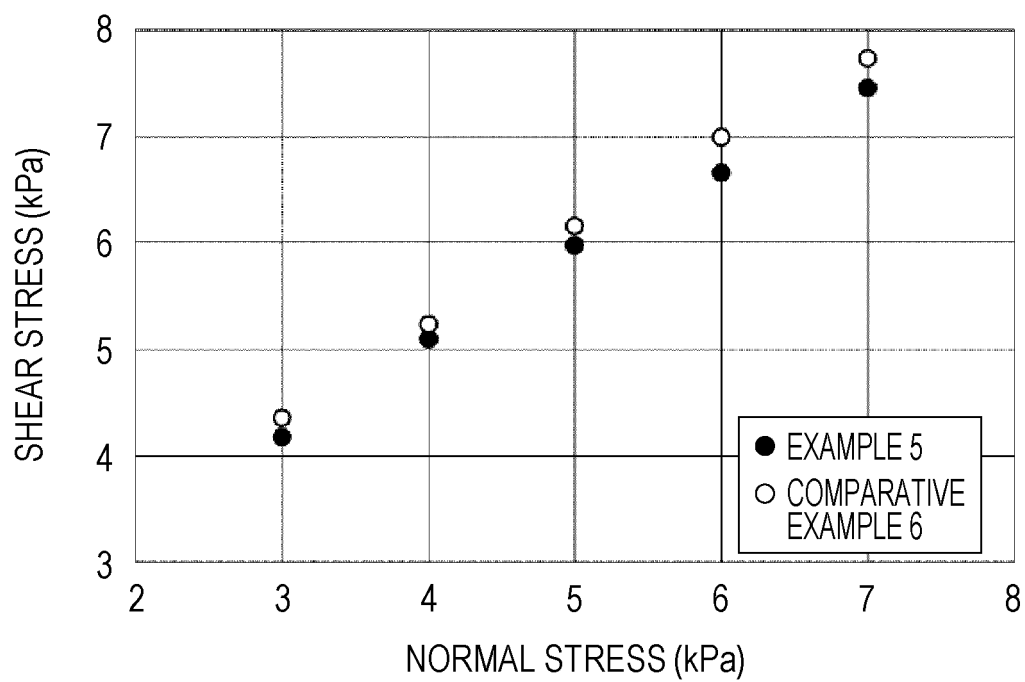
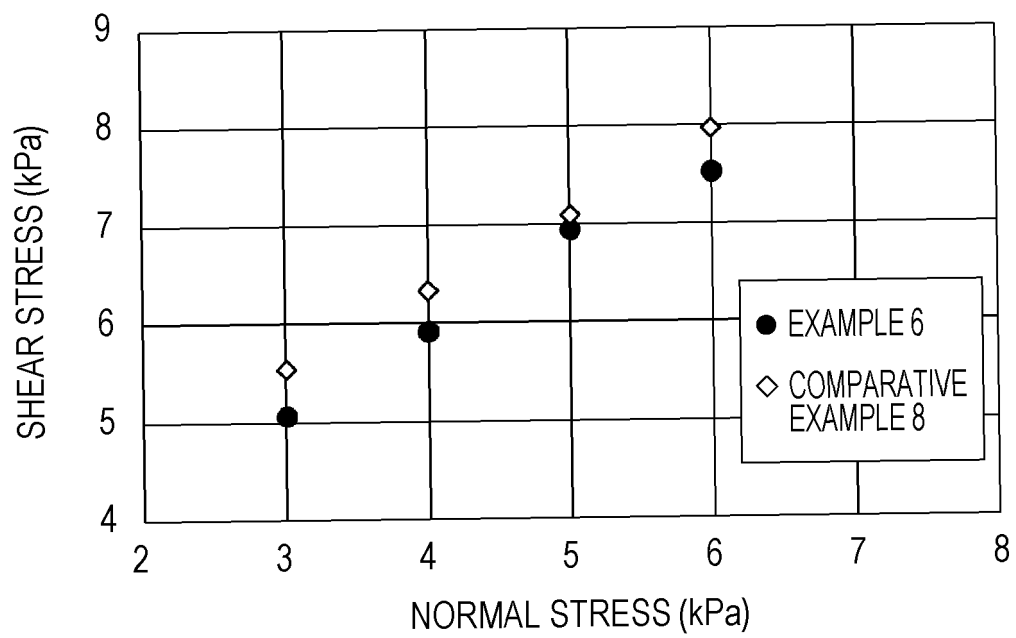


FIG. 7



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INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2021/016101

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A. CLASSIFICATION OF SUBJECT MATTER

Int.Cl. A24B13/00 (2006.01) i, A24B15/30 (2006.01) i

FI: A24B13/00, A24B15/30

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

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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Int.Cl. A24B13/00, A24B15/30

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

Published examined utility model applications of Japan	1922-1996
Published unexamined utility model applications of Japan	1971-2021
Registered utility model specifications of Japan	1996-2021
Published registered utility model applications of Japan	1994-2021

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

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Y	December 2009 (2009-12-24), paragraphs [0017]-[0057], fig. 1	8-9
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Y	US 2010/0303969 A1 (PHILIP MORRIS USA INC.) 02 December 2010 (2010-12-02), paragraph [0079]	9

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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Date of the actual completion of the international search
03 June 2021Date of mailing of the international search report
15 June 2021

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

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paragraphs [0064], [0065]
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US 2010/0303969 A1 02 December 2010 (Family: none)

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Form PCT/ISA/210 (patent family annex) (January 2015)

REFERENCES CITED IN THE DESCRIPTION

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