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(71) Applicant: **Swedish Match North Europe AB**
118 85 Stockholm (SE)

(72) Inventor: **BODIN, Aase**
417 62 Göteborg (SE)

(74) Representative: **Valea AB**
Box 1098
405 23 Göteborg (SE)

(54) **A FLAVOURED ORAL POUCHED NICOTINE PRODUCT COMPRISING AN ACID**

(57) There is disclosed an oral pouched nicotine product comprising a filling material and a saliva-permeable pouch of a packaging material enclosing the filling material,
the filling material comprising:
- a non-tobacco material,
- a nicotine source,
- a flavouring agent,
- moisture in an amount within the range of from 1 wt % to 50 wt% based on the total weight of the filling material,
- a pH adjuster comprising one or more of the following: Na₂CO₃, K₂CO₃, MgCO₃, NaHCO₃, KHCO₃, NaOH,

KOH, and
- optionally a tobacco material within the range of from 0.05 wt% to 5 wt% based on the total weight of the filling material,
the packaging material comprising an acid selected from the group consisting of citric acid, malic acid, lactic acid, ascorbic acid, tartaric acid and any combination thereof, said acid being present in an amount within the range of from 5 wt % to 20 wt% based on the total dry weight of the packaging material.
There is also provided a method for manufacture of the oral pouched nicotine product.

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Description

TECHNICAL FIELD

5 **[0001]** The present disclosure relates to a tobacco free or low tobacco oral pouched nicotine product comprising a filling material and a saliva-permeable pouch of a packaging material enclosing the filling material. The filling material comprises nicotine and a flavourant, and the packaging material comprises an acid selected from the group consisting of citric acid, malic acid, lactic acid, ascorbic acid, tartaric acid, and any combination thereof. Upon oral administration of the oral pouched nicotine product the acid of the packaging material is released to provide a sour sensation which is followed by release of the flavourant together with the nicotine of the filling material.

BACKGROUND

15 **[0002]** Moist snuff for oral use is available in loose form or portion-packed in a saliva-permeable, porous wrapper material forming a pouch. Pouched moist snuff is typically used by the user by placing the pouch between the upper or lower gum and the lip or cheek and retaining it there for a limited period of time. The pouch material holds the tobacco in place while allowing saliva to pass into the interior of the pouched product and allowing flavours and nicotine to diffuse from the tobacco material into the user's mouth.

20 **[0003]** There are also tobacco-free or substantially tobacco-free oral pouched nicotine-containing products available which may be offered as alternatives to oral pouched smokeless tobacco products. These tobacco-free or substantially tobacco-free oral pouched nicotine-containing products are generally used in the same manner as the oral pouched tobacco-containing products and are herein referred to as oral pouched nicotine products.

25 **[0004]** Oral pouched smokeless tobacco products as well as tobacco-free or substantially tobacco-free oral pouched nicotine products may be produced by measuring portions of the filling material and inserting the portions into a packaging material. The packaging material forming the pouch in oral pouched products is typically a dry-laid bonded nonwoven comprising viscose rayon fibres (i.e. regenerated cellulose) and an acrylic polymer that acts as binder in the nonwoven material and provides for heat-sealing of the pouches during manufacturing thereof. The packaging material forming the pouch of the oral pouched product should during manufacturing of the pouch provide for sealing, upon storage of the pouch exhibit none or a low degree of discoloration and upon usage by a consumer preserve integrity and strength, allow for a desired release profile of nicotine and flavours and provide a pleasant mouth-feel.

30 **[0005]** The organoleptic properties, such as texture, aroma, taste, shape and appearance, of the oral pouched product are of high importance to the user. It is generally desirable to provide oral pouched nicotine products with rapid release of flavour and/or nicotine to provide an initial strong flavour experience and/or reduce nicotine craving. However, there is generally a lag time before onset of the extraction of the flavour and nicotine from the filling material of the oral pouched product takes place in the oral cavity. This is perceived as unsatisfactory by many consumers who desire an immediate effect upon intake of the oral pouched nicotine product.

35 **[0006]** Moreover, many consumers are fond of a sour taste, i.e. acidity. However, acids may protonate the nicotine thereby decreasing its uptake through the mucous membranes in the oral cavity and/or react with basic components such as basic pH adjusters in the filling material.

40 **[0007]** Thus, there is a need for an oral pouched nicotine product allowing for release such as fast release of sour taste. Further, there is a need for an oral pouched nicotine product allowing for release sour taste without or substantially without negatively impacting release of the nicotine.

SUMMARY

45 **[0008]** An object of the present disclosure is to alleviate at least one of the problems discussed above, and/or to provide advantages and aspects not provided by hitherto known technique.

[0009] One or more of the above objects may be achieved by the oral pouched nicotine product according to appended claim 1, and/or by the method according to appended claim 14.

50 **[0010]** Thus, the present disclosure provides an oral pouched nicotine product comprising a filling material and a saliva-permeable pouch of a packaging material enclosing the filling material, the filling material comprising:

- a non-tobacco material,
- a nicotine source,
- 55 - a flavouring agent,
- moisture in an amount within the range of from 1 wt % to 50 wt% based on the total weight of the filling material,
- a pH adjuster comprising one or more of the following: Na₂CO₃, K₂CO₃, MgCO₃, NaHCO₃, KHCO₃, NaOH, KOH, and
- optionally a tobacco material within the range of from 0.05 wt% to 5 wt% based on the total weight of the filling material,

the packaging material comprising an acid selected from the group consisting of citric acid, malic acid, lactic acid, ascorbic acid, tartaric acid and any combination thereof, said acid being present in an amount within the range of from 5 wt % to 20 wt% based on the total dry weight of the packaging material.

[0011] Further, the present disclosure also provides a method for preparing an oral pouched nicotine product as described herein, the method comprising the steps of:

- a) providing a filling material as described herein, and
- b) enclosing the filling material in the packaging material obtained in step a).

DEFINITIONS

[0012] The term "tobacco material" is used herein for fibrous material of tobacco leaves or parts of leaves, such as lamina and stem. The leaves and parts of leaves may be finely divided (disintegrated), such as ground, cut, shredded or threshed, and the parts of leaves may be blended in defined proportions in the tobacco material. The tobacco material may comprise or be free from tobacco extract.

[0013] By "tobacco" as used herein is meant any part, e.g., leaves, stems, and stalks, of any member of the genus *Nicotiana*. The tobacco may be whole, shredded, threshed, cut, ground, cured, aged, fermented, or treated otherwise, e.g., granulated or encapsulated.

[0014] "Oral" and "oral use" is in all contexts used herein as a description for use in the oral cavity of a human, such as buccal placement.

[0015] As used herein, the term "moisture content" refers to the total amount of oven volatile ingredients, such as water and other oven volatiles (e.g. propylene glycol) in the preparation, composition or product referred to. The moisture content is given herein as percent by weight (wt%) of the total weight of the preparation, composition or product referred to.

[0016] The moisture content as referred to herein may be determined by using a method based on literature references Federal Register/ vol.74, no. 4/712-719/Wednesday, January 7, 2009/Notices "Total moisture determination" and AOAC (Association of Official Analytical Chemists), Official Methods of Analysis 966.02: "Moisture in Tobacco" (1990), Fifth Edition, K. Helrich (ed). In this method, the moisture content is determined gravimetrically by taking 2.5 ± 0.25 g sample and weighing the sample at ambient conditions, herein defined as being at a temperature of 22°C and a relative humidity of 60%, before evaporation of moisture and after completion of dehydration. Mettler Toledo's Moisture Analyzer HB43, a balance with halogen heating technology, is used (instead of an oven and a balance as in the mentioned literature references) in the experiments described herein. The sample is heated to 105°C (instead of 99.5 ± 0.5 °C as in the mentioned literature references). The measurement is stopped when the weight change is less than 1 mg during a 90 seconds time frame. The moisture content as weight percent of the sample is then calculated automatically by the Moisture Analyzer HB43.

[0017] Some fibrous materials may exhibit hygroscopic properties. Hygroscopic materials maintain equilibrium moisture content depending on the ambient moisture and temperature.

[0018] "Flavour", "flavourant" or "flavouring agent" is used herein for a substance used to influence the aroma and/or taste of the nicotine product, including, but not limited to, essential oils, single flavour compounds, compounded flavourings, and extracts.

[0019] As used herein "% w/w" or "wt%" or "weight %" or "% by weight" refers to the weight percent of the ingredient referred to of the total weight of the preparation, composition or product referred to. In this document, the expressions "per cent by weight", weight% and wt% are used interchangeably.

[0020] As used herein, reference to "dry weight percent", "% by weight, based on dry weight" and the like refers to the weight percent of the ingredient referred to on the basis of the total weight of the dry ingredients, i.e. all ingredients of the preparation, composition or product referred to excluding the moisture content.

[0021] As used herein, reference to "wet weight percent", "% by weight, based on wet weight" and the like refers to the weight percent of the ingredient referred to on the basis of the total weight of the ingredients, i.e. all ingredients of the preparation, composition or product referred to including the moisture content. Thus, "% by weight, based on total weight" as used herein is the same as "% by weight, based on wet weight".

[0022] As used herein the terms "pouched nicotine product for oral use" or "oral pouched nicotine product" refer to a portion of nicotine-containing filling material packed in a saliva-permeable pouch material intended for oral use.

[0023] As used herein the terms "oral pouched nicotine non-tobacco product", "oral pouched nicotine product free from tobacco" or "oral pouched tobacco-free product" refer to a portion of nicotine-containing filling material packed in a saliva-permeable pouch material intended for oral use wherein no tobacco is included in said product.

[0024] As used herein the terms "oral pouched nicotine tobacco product" or "low tobacco oral pouched nicotine product" refer to a portion of nicotine-containing filling material packed in a saliva-permeable pouch material intended for oral use wherein an amount of tobacco material within the range of from about 0.1 % to about 10% by weight or from about 0.1% to about 5% by weight, based on the total weight of the filling material, is included in said product.

[0025] As used herein, the term "particulate" refers to a component in the form of particles, including granules, pellets, powder, etc.

[0026] As used herein, the term "fibrous" refers to a component which is constituted by natural or man-made fibers or which is substantially constituted by natural or man-made fibers.

5 **[0027]** As used herein, the term "non-particulate" refers to a component which is not in the form of particle(s).

DESCRIPTION

10 **[0028]** The present disclosure provides an oral pouched nicotine product comprising a filling material and a saliva-permeable pouch of a packaging material enclosing the filling material, the filling material comprising:

- a non-tobacco material,
- a nicotine source,
- 15 - a flavouring agent,
- moisture in an amount within the range of from 1 wt % to 50 wt% based on the total weight of the filling material,
- a pH adjuster comprising one or more of the following: Na₂CO₃, K₂CO₃, MgCO₃, NaHCO₃, KHCO₃, NaOH, KOH, and
- optionally a tobacco material within the range of from 0.05 wt% to 5 wt% based on the total weight of the filling material,

20 the packaging material comprising an acid selected from the group consisting of citric acid, malic acid, lactic acid, ascorbic acid, tartaric acid and any combination thereof, said acid being present in an amount within the range of from 5 wt % to 20 wt% based on the total dry weight of the packaging material.

[0029] The oral pouched nicotine product may be free from tobacco, i.e. it does not contain tobacco, thereby providing an oral pouched nicotine tobacco-free product. Alternatively, the oral pouched nicotine product may comprise a small amount of tobacco material, such as from about 0.1% by weight to about 10% by weight based on the total weight of the filling material, thereby providing a low tobacco oral pouched nicotine product. For example, the low tobacco oral pouched nicotine product may comprise a filling material comprising from 0.1% by weight to about 10% by weight, such as from about 0.1% by weight to about 5% by weight, such as from about 0.2% by weight to about 1% by weight, such as from 0.1 wt% to 1 wt%, of tobacco material based on the total weight of the filling material. In particular, the low tobacco oral pouched nicotine product may comprise a filling material comprising from about 0.2% by weight to about 1% by weight, such as about 0.2 % by weight, of tobacco material based on the total weight of the filling material. It will be appreciated that the tobacco material in the low tobacco oral nicotine product described herein forms part of the filling material.

35 **[0030]** The non-tobacco material may be any suitable material which can act as a bulk material in the filling material to provide the oral pouched product with a desired pre-use volume. The non-tobacco material may further serve as a substrate for one or more other components in the filling material.

[0031] The non-tobacco material may be a particulate material, i.e., a material in the form of particles of any suitable size or shape, including granules, powders, pellets, irregularly shaped particles, spherical particles, etc.

40 **[0032]** The non-tobacco material may be in the form of fibers, such as natural fibers or man-made fibers. Mixtures of fibers and particles are also contemplated for the non-tobacco materials as disclosed herein.

[0033] The non-tobacco material may be provided in the form of a paste or coherent mass.

[0034] The non-tobacco material may be water-insoluble, water-soluble or a mixture thereof. For instance, the non-tobacco material comprises a sugar alcohol such as maltitol and/or cellulose such as microcrystalline cellulose and/or powdered cellulose. In an example, the non-tobacco material may comprise maltitol and microcrystalline cellulose.

45 **[0035]** The nicotine source of the filling material may be one or more of the following: nicotine base, nicotine hydrochloride, nicotine dihydrochloride, nicotine monotartrate, nicotine bitartrate, nicotine bitartrate dihydrate, nicotine sulphate, nicotine zinc chloride monohydrate and nicotine salicylate, nicotine benzoate, nicotine polacrilex. In particular, the moist filling material may comprise or consist of one or more of the following: nicotine bitartrate, nicotine bitartrate dihydrate, an aqueous solution comprising nicotine base and tartaric acid such as an aqueous solution comprising about 20% by weight of nicotine base and about 10% by weight of tartaric acid, based on the total weight of the aqueous solution.

50 **[0036]** It will be appreciated that the nicotine source described herein does not comprise tobacco such as tobacco material.

[0037] The filling material may comprise the nicotine source in an amount within a range of from about 0.5 wt% to about 10 wt%, such as from about 0.2 wt% to about 3 wt%, calculated as nicotine base, based on the total weight of the moist filling material.

55 **[0038]** The amount of nicotine source per pouched product may be within the range from about 0.1 mg to about 30 mg of nicotine calculated as nicotine base, such as about 0.5 mg, about 1.0 mg, about 1.5 mg, about 2.0 mg, about 2.5 mg, about 3.0 mg, about 3.5 mg, about 4.0 mg, about 4.5 mg, about 5.0 mg, about 6.0 mg, about 7.0 mg, about 8.0 mg,

about 9.0 mg, about 10 mg, about 11 mg, about 12, about 13 mg, about 14, about 15 mg, about 16 mg, about 17 mg, about 18 mg, about 19 mg, about 20 mg, about 21 mg, about 22 mg, about 23 mg, about 24 mg or about 25 mg of nicotine. As used herein, mg stands for milligram(s).

5 **[0039]** The filling material comprises one, two or more flavouring agent(s). The flavouring agent(s) may be non-encapsulated and/or encapsulated. As used herein, an encapsulated flavouring agent is a flavouring agent contained within a capsule. Accordingly, a non-encapsulated flavouring agent is not contained within a capsule. The flavourant may comprise or consist of a flavour oil, such as a hydrophobic flavour oil, such as a synthetic flavour, such as a nature-identical flavour. For instance, the flavouring agent of the filling material in the oral pouched nicotine product as disclosed herein may comprise or consist of one or more of the following: a synthetic flavour such as a nature-identical flavour, a plant based flavour, a flavour oil, a hydrophobic flavour oil such as an essential oil. As used herein, a nature-identical flavour intends a synthetic flavour which is chemically identical to natural flavourings but are prepared or extracted using chemical methods. The flavouring agent may be a mixture of different flavours. The flavouring agent may be provided as an oil such as a hydrophobic oil, a liquid, a powder or a mixture thereof. Further, the flavouring agent(s) may be in the form of a liquid and/or a solid. The flavouring agent may be stable at pH > 7.

10 **[0040]** Examples of flavours of the flavouring agent(s) include bergamot, eucalyptus, orange, mandarin, citrus, lemon, peppermint, spearmint, mint, menthol, liquorice, wintergreen, whiskey, rum, cherry, various berries, tobacco, coffee, vanilla, lime, apple, peach, carvone, limonene and any combination of two or more thereof. For instance, the flavouring agent may be one or more of the following: lemon oil, lime oil, orange oil, grapefruit oil, bergamot oil.

15 **[0041]** The moist filling material may further comprise an encapsulated flavouring agent. The encapsulated flavouring agent may be the same or different from the non-encapsulated flavouring agent. Alternatively, the moist filling material may be free from encapsulated flavouring agent.

20 **[0042]** The moist filling material of the oral pouched nicotine product as disclosed herein may comprise within the range of from about 0.5% to about 5.0% by weight, such as from 0.5% by weight to about 3 % by weight, of the flavouring agent, based on the total weight of the moist filling material.

25 **[0043]** The filling material of the oral pouched nicotine product described herein may further comprise a sweetener such as a natural or artificial sweetener. The artificial sweetener may comprise acesulfame potassium. The sweetener may be present in an amount within the range of from about 0.1% w/w to about 1% w/w based on the total weight of the moist filling material.

30 **[0044]** Further, the filling material of the oral pouched nicotine product described herein may further comprise a salt selected from the group consisting of sodium chloride, potassium chloride, magnesium chloride, calcium chloride and any combination of two or more thereof. In particular, the salt may comprise or consist of sodium chloride. The salt may be present in an amount within the range of from about 1.0% w/w to about 10% w/w, such as from about 2.5% w/w to about 5% w/w, based on the total weight of the filling material.

35 **[0045]** The filling material of the oral pouched nicotine product described herein may have a moisture content within the range of from about 10% to about 60% by weight, such as from about 40% to about 60% by weight, such as from about 35% to about 55% by weight, such as about 35% to about 45% by weight, such as about 30% to about 40% by weight, such as from about 50% to about 60% by weight, based on the total weight of the moist filling material. In an example, the moisture of the filling material may be present in an amount from about 20 wt% to about 50 wt%, such as from about 20 wt% to about 45 wt%, such as about 40 wt%, based on the total weight of the filling material. In still a further example, the moisture of the filling material may be present in an amount within the range of from 1 wt% to 20 wt%, such as from 15 wt% to 20 wt%, such as from 12 wt% to 20 wt%, 1 wt% to 12 wt%, such as from 1 wt% to 5 wt%, such as about 3 wt%, such as from 0.1 wt% to 1 wt%, based on the total weight of the filling material.

40 **[0046]** The moisture of the filling material described herein may be provided by water and optionally a humectant. The humectant may comprise or consist of glycerol and/or propylene glycol. The humectant may be present in an amount within the range of from about 5% w/w to about 15% w/w, based on the total weight of the filling material.

45 **[0047]** The pH adjuster of the filling material described herein may comprise or consist of one or more of the following: Na₂CO₃, K₂CO₃, MgCO₃, NaHCO₃, KHCO₃, NaOH, KOH. For instance, the pH adjuster may comprise or consist of Na₂CO₃ and optionally NaHCO₃ and/or KOH. It will be appreciated that the filling material may be free from an acid such as an acid described herein. Alternatively, the filling material may comprise a small amount of acid such as tartaric acid. The acid may be present in an amount that does not negatively impact the pH of the filling material. Further, the acid such as tartaric acid may be present in combination with nicotine thereby forming a nicotine salt. The amount of pH adjusting agent may be selected such that the moist filling material when dispersed in purified water provides a pH above 7.0, such as a pH within the range of from about 7.0 to about 10.0 or a pH within the range of from about 8.0 to about 9.0, such as a pH within the range of from about 8.3 to about 8.7. For example, the pH adjusting agent(s) may be present in an amount of from about 1.0% to about 15% by weight, based on total weight of the moist filling material.

50 **[0048]** The pH of the moist filling material may be measured by adding 100 ml of distilled water to 5.0 gram of moist filling material, for instance in a 100 ml Erlenmeyer flask, stirring the resulting mixture at room temperature with a magnetic stirrer at 100 rpm for about 5 minutes, and then measuring the pH of an extract obtained therefrom with a calibrated

(according to the manufacturer's instructions) pH meter. For correctness of readings, the sample solutions shall be analyzed within one hour. In this document, the term "rpm" stands for revolutions per minute. Further, in this document the expression "room temperature" stands for from about 20°C to about 25°C such as about 22°C.

5 **[0049]** The packaging material of the oral pouched nicotine product described herein may comprise or consist of a saliva-permeable nonwoven material. The nonwoven material may be dry-laid, wet-laid or spunbond. The nonwoven material may comprise fibers and/or binder(s) allowing for sealing such as heat-sealable fibers. For instance, the binder(s) may comprise acrylate binders. Thus, the pouch of the oral pouched nicotine product described herein may be formed by heat sealing. Additionally or alternatively, the pouch may be formed by ultrasonic welding to provide ultrasonic seals.

10 **[0050]** The packaging material of the oral pouched nicotine product described herein comprises an acid selected from the group consisting of citric acid, malic acid, lactic acid, ascorbic acid, tartaric acid and any combination thereof. In particular, the acid may comprise or consist of citric acid. It will be appreciated that the acid included in the packaging material may be provided as a salt thereof. Alternatively, the acid included in the packaging material is not provided as a salt.

15 **[0051]** Surprisingly, it has been found that the acid of the packaging material substantially does not migrate into the filling material upon storage. Instead, the acid of the packaging material remains in the packaging material and allows for a fast release of the acid thereby providing sour taste to the oral cavity of a consumer. In this way, the consumer can enjoy a fast release of the acid followed by a slower release of the flavouring agent and nicotine of the filling material. Thus, the consumer will experience an initial fast release of acid from the oral pouched nicotine product and also an overall longer organoleptic experience. Additionally, the limited contact between the acid in the packaging material and the filling material minimizes the risk that the acid will interact with components in the filling material to provide e.g. protonation of the nicotine leading to reduced oral uptake of the nicotine and/or neutralization of basic components such as pH adjusters in the filling material. Surprisingly, it has also been found that the presence of an acid in the packaging material substantially does not negatively impact the seal strength of the packaging material.

20 **[0052]** The acid in the packaging material may be present in an amount within the range of from 5 wt% to about 20 wt%, from about 5 wt% to about 15 wt%, from about 10 wt% to about 15 wt% or from about 5 wt% to about 10 wt%, based on the total weight of the packaging material. In particular, the acid in the packaging material may be present in an amount within the range of from about 10 wt% to about 15 wt%, such as about 12 wt%, based on the total weight of the packaging material.

25 **[0053]** There is also provided a method for manufacturing the oral pouched nicotine product described herein, the method comprising the steps of:

- 30
- a) providing a filling material as described herein, and
 - b) enclosing the filling material in the packaging material obtained in step a).

35 **[0054]** The packaging material described herein may be produced by

- 40
- (i) treatment of a packaging material with an aqueous solution comprising an acid selected from the group consisting of citric acid, malic acid, lactic acid, ascorbic acid, tartaric acid and any combination thereof, followed by
 - (ii) drying. The aqueous solution may comprise or consist of water and optionally a food grade solvent such as ethanol. The packaging material may be a nonwoven material as described herein. The treatment of step (i) may take place by immersing such as dipping the packaging material into the aqueous solution. Additionally, or alternatively, the treatment of step (i) may take place by spraying the aqueous solution onto the packaging material. The drying step (ii) may take place at room temperature or at a temperature exceeding room temperature.

45 **[0055]** The disclosure is illustrated by means of the following non-limitative Examples.

EXAMPLES

General

50 **[0056]** The dry-laid and saliva-permeable nonwoven materials NW37 and Z8732 were used in the following examples. NW37 was as described in US 2020/0297024 A1, paragraph [0101], except that the basis weight was 37 g/m² instead of 38 g/m². Z8723 was purchased from Tenowo, Germany, and included polyethylene terephthalate fibers and chemical binder. Citric acid was purchased from Kockens, Sweden, and VWR, Sweden, respectively. The citric acid from VWR was provided as a monohydrate. DL-Malic acid, L-(+)-lactic acid, and L-ascorbic acid and L-(+)-tartaric acid were purchased from Sigma-Aldrich, Sweden.

Measurement of pH

5 [0057] The pH of the filling material was measured as follows. 100 ml of distilled water was added to 5.0 gram of filling material followed by stirring the resulting mixture at room temperature with a magnetic stirrer at 100 rpm for about 5 minutes, and then measuring the pH of an extract obtained therefrom with a calibrated (according to the manufacturer's instructions) pH meter. For correctness of readings, the sample solutions shall be analyzed within one hour. In this document, the term "rpm" stands for revolutions per minute. Further, in this document the expression "room temperature" stands for from about 20°C to about 25°C such as about 22°C. The pH of the pouch was measured in the same way as for the filling material, but instead of the filling material a mixture of the filling material and the nonwoven pouch material was used.

Measurement of pouch seal strength

15 [0058] The pouch seal strength was measured using an Instron 5943 instrument as follows. One ply is attached to the upper gauge and one ply to the lower gauge. The force used to peel apart the seal was determined and expressed as load per width at maximum load (Newton per millimeter(s), i.e. N/mm). The following machine parameters were used:

load range: 50 N

20 extension: 10 mm

gauge length: 13 mm

25 speed: 10 mm/min

preload: 0.1 N

sample width: 12 mm

30 Measurement of Nonwoven seal strength

[0059] The seal strength of the nonwoven material was measured using an Instron 5943 instrument as follows. The nonwoven material was heat-sealed in accordance with EP3192380A to provide a seal. One ply was attached to the upper gauge and one ply to the lower gauge. The force used to peel apart the seal was determined and expressed as load per width at maximum load (Newton per millimeter(s), i.e. N/mm). As used herein, N stands for Newton, mm stands for millimeter(s) and min stands for minute(s). The following machine parameters were used:

load range: 50N

40 extension: 10mm

gauge length: 13mm

45 speed: 30mm/min

preload: 0.1N

sample width: 33-43mm

50 Example 1A

[0060] Citric acid was dissolved in water to provide aqueous solutions comprising 1 wt%, 2 wt%, 3 wt%, 4 wt%, 5 wt% and 6 wt% of citric acid, based on the total weight of the aqueous solution. The citric acid was included in the nonwoven materials NW37 and Z8732 as follows. The nonwoven material was dipped into the aqueous solution to become completely wet and was then instantaneously removed. Thereafter, the nonwoven material was dried at room temperature for 30 minutes or longer if necessary, to become dry.

[0061] The concentration of citric acid included in the nonwoven material was calculated by measuring the weight of the nonwoven material before it was treated with the aqueous solution comprising citric acid and after it had been treated

with the citric acid aqueous solution followed by drying and using equation 1 below:

$$\% \text{ concentration} = \frac{X-Y}{Y} \times 100\% \quad \text{Equation 1}$$

In Equation 1, X is the weight of the nonwoven material after it has been treated with the citric acid aqueous solution and dried, and Y is the weight of the nonwoven before it has been treated with the citric acid aqueous solution. Thus, the concentration is the amount of citric acid in the dried nonwoven material after treatment with the citric acid aqueous solution. The concentration of citric acid included in the nonwoven material may also be denoted the amount of citric acid included in the nonwoven material.

[0062] Table 1 shows the concentration of citric acid included in the dried nonwoven material treated with the citric acid aqueous solutions.

Table 1:

Concentration of citric acid aqueous solution (wt%)	NW37 Concentration of included citric acid (wt%)	Z8732 Concentration of included citric acid (wt%)
1	4	3
2	7	5
3	11	7
4	15	11
5	20	13
6	24	16

[0063] The peel strength was measured for the dried nonwoven materials treated with the citric acid aqueous solutions, which gave the results shown in Table 2.

Table 2:

Concentration of citric acid aqueous solution (wt%)	Peel strength for NW37 (N/mm)	Peel strength for Z8732 (N/mm)
1	0.20	0.28
2	0.17	0.22
3	0.16	0.23
4	0.14	0.17
5	0.18	0.19
6	0.17	0.17

Example 1B

[0064] Two people tasted oral pouches comprising the nonwoven material NW37 treated with citric acid from Example 1A in their mouths and found that the NW37 treated with citric acid in concentrations of 1 wt% and 2 wt% gave no or very little acidity. However, NW37 treated with citric acid in concentrations of 3 wt%, 4 wt% and 5 wt% were found to give a pleasant acidity. These concentrations correspond to 11 wt%, 15 wt% and 20 wt% of citric acid in the NW37, based on the total weight of the dried NW37. It was concluded that nonwoven material comprising from 11 wt% to 20 wt% of citric acid based on the total weight of the dried nonwoven material gave a pleasant acidity.

Example 1C

[0065] This example was performed in the same way as Example 1A, but the citric acid was dissolved in ethanol (96% in water) instead of water. In this way, the drying at room temperature was faster compared to Example 1A. The concentration of included citric acid in the nonwoven material and the peel strength were measured and the results are shown in Tables 3 and 4, respectively.

Table 3:

Concentration of citric acid ethanol solution (wt%)	NW37 Concentration of included citric acid (wt%)	Z8732 Concentration of included citric acid (wt%)
1	4	4
2	10	7
3	17	12
4	21	15
5	29	20
6	32	25

[0066] Comparison of the results in Table 3 with the results in Table 1 shows that the concentration of citric acid incorporated in the nonwoven materials generally was higher when ethanol was used as solvent instead of water.

Table 4:

Concentration of citric acid in ethanol solution (wt%)	Peel strength for NW37 (N/mm)	Peel strength for Z8732 (N/mm)
1	0.18	0.28
2	0.18	0.25
3	0.17	0.22
4	0.17	0.23
5	0.14	0.22
6	0.14	0.22

Example 2

[0067] A first filling material comprising nicotine, microcrystalline cellulose, maltitol, lemon flavourant, sodium chloride, pH adjuster and moisture was provided. The moisture content was 3 wt% based on the total weight of the filling material. A second filling material was also provided, said second filling material being substantially the same as the first filling material but having a moisture content of 40 wt% based on the total weight of the filling material.

[0068] The first filling material was packed into portion pouches with ultrasonic seals of 0.4 g each using the nonwoven material NW37 treated with an aqueous solution comprising 3.5 wt% of citric acid. The concentration of citric acid incorporated in the nonwoven material NW37 was 12.5 wt% based on the total weight of the dried NW37.

[0069] The second filling material was packed into portion pouches with heat-sealed seals of 0.9 g each using the nonwoven material Z8732 treated with an aqueous solution comprising 4.5 wt% of citric acid. The concentration of citric acid incorporated in the nonwoven material Z8732 was 12.5 wt% based on the total weight of the dried Z8732.

[0070] The pouches were packed into plastic cans suitable for containment of oral pouches. The plastic cans were as described in WO 2017/125405. The filling material pH and the pouch seal strength were analysed before and after storage in a climate cabinet (VC0100, Vötsch Industrietechnik) for storage. The cabinet condition was set at 0°C, which

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corresponds to refrigerator conditions. For some samples, storage in the climate cabinet was followed by storage at rt (i.e. room temperature) The pH and the pouch seal strength were measured as described above. The results are shown in Tables 5 and 6. A comparative experiment is shown in Table 7.

Table 5: Pouches comprising filling material 1 and the nonwoven material NW37 comprising 12.5 wt% of citric acid

Sample No.	Storage time and condition (s)	pH of filling material	pH of pouch	Peel strength (N/mm)
1	0 weeks	8.2	6.7	0.18
2	1 week refrigerated	8.2	6.6	0.18
3	1 week refrigerated + 5 weeks at rt	8.1	6.6	0.16

Table 6: Pouches comprising filling material 2 and the nonwoven material Z8732 comprising 12.5 wt% of citric acid

Sample No.	Storage time and condition(s)	pH of filling material	pH of pouch
1	1 week refrigerated	8.4	7.8
2	1 week refrigerated + 3 weeks at rt	8.3	8.1

Table 7: Comparative example. Pouches comprising filling material 2 and the nonwoven material Z8732

Sample No.	Storage time and condition(s)	pH of filling material	pH of pouch
<u>1</u>	1 week refrigerated	8.8	8.7
<u>2</u>	1 week refrigerated + 3 weeks at rt	8.7	8.7

[0071] As shown in Table 5, for the pouches comprising filling material 1, said filling material having a moisture content of about 3 wt%, storage did not substantially affect the pH of the filling material or the pH of the pouch (i.e. the pH measured on a sample comprising the filling material and the nonwoven material). Further, Table 5 shows that the pouch seal strength as measured by the peel strength was substantially unaffected by the storage. It was concluded that the citric acid remained in the nonwoven material, i.e. the citric acid did not or substantially did not migrate into the filling material during storage since this would have lowered the pH of the filling material.

[0072] As shown in Table 6, for the pouches comprising filling material 2, said filling material having a moisture content of about 40 wt%, storage did not substantially affect the pH with respect to the filling material and the pouch. It was concluded that the citric acid remained in the nonwoven material, i.e. the citric acid did not substantially migrate into the filling material during storage since this would have lowered the pH of the filling material. Further, comparison of the results in Table 6 with a pouch lacking citric acid as shown in Table 7 shows that the presence of citric acid in the nonwoven pouch material provides a product with lower pH with respect to the filling material and the pouch. Thus, it appears that the presence of citric acid lowers the pH when it is prepared. However, once prepared the pH remains substantially unchanged upon storage.

Example 3

[0073] 4 people tested oral pouched products comprising (i) the nonwoven material NW37 comprising the amounts of citric acid as described in Example 1A and (ii) the filling material 1 as described in Example 2. It was found that the pouched product comprising the nonwoven material NW37 comprising 12.5 wt% of citric acid, based on the total weight of the NW37, gave an initial and appealing acidity followed by lemon flavour from the filling material.

[0074] In the same way, testing was performed for oral pouched products comprising (i) the nonwoven material Z8732 comprising various amounts of citric acid as described in Example 1A and (ii) the filling material 2 as described in Example 2. It was found that the acidity remained after storage in refrigerator for 1 week and also after storage in refrigerator for 1 week followed by storage at room temperature for 3 weeks.

Example 4

[0075] The nonwoven material NW37 was treated with an acid selected from the group consisting of malic acid, lactic acid, ascorbic acid and tartaric acid in the same way as described in Example 1A. The dried NW37 contained about 12 wt% to 14 wt% of the acid. Thus, each of malic acid, lactic acid, ascorbic acid and tartaric acid was separately used instead of citric acid.

[0076] Pouched products comprising the nonwoven material NW37 comprising the acid and the filling material 1 as described in Example 2 were prepared, together with a reference example lacking acid, and tested by 4 people. For each of the four acids it was found that the pouched products gave an initial acidity followed by lemon flavour. This initial acid flavour was lacking in the reference sample.

[0077] The peel strength was tested for the nonwoven material NW37 comprising the acids, and also for a reference sample lacking acid. The results are shown in Table 8.

Table 8:

Sample No.	Pouch material	Peel Strength (N/mm)
Reference	NW37	0.18
1	NW37 comprising ascorbic acid	0.19
2	NW37 comprising malic acid	0.20
3	NW37 comprising lactic acid	0.19
4	NW37 comprising tartaric acid	0.11

[0078] It was concluded that the seal strength of the pouch material in Samples No. 1-3 was not substantially affected by the presence of an acid. In contrast, the seal strength was lowered for Sample. No 4.

References**[0079]**

1. Federal Register/ vol.74, no. 4/712-719/Wednesday, January 7, 2009/Notices "Total moisture determination.
2. Official Methods of Analysis 966.02: "Moisture in Tobacco" (1990), Fifth Edition, K. Helrich (ed).
3. US 2020/0297024 A1
4. EP3192380A
5. WO 2017/125405

Claims

1. An oral pouched nicotine product comprising a filling material and a saliva-permeable pouch of a packaging material enclosing the filling material, the filling material comprising:
 - a non-tobacco material,
 - a nicotine source,
 - a flavouring agent,
 - moisture in an amount within the range of from 1 wt % to 50 wt% based on the total weight of the filling material,
 - a pH adjuster comprising one or more of the following: Na₂CO₃, K₂CO₃, MgCO₃, NaHCO₃, KHCO₃, NaOH, KOH, and
 - optionally a tobacco material within the range of from 0.05 wt% to 5 wt% based on the total weight of the filling material,

the packaging material comprising an acid selected from the group consisting of citric acid, malic acid, lactic acid, ascorbic acid, tartaric acid and any combination thereof, said acid being present in an amount within the range of from 5 wt % to 20 wt% based on the total dry weight of the packaging material.

- 5 **2.** The oral pouched nicotine product according to claim 1, wherein the oral pouched nicotine product is free from tobacco or wherein the tobacco material is present in an amount within the range of from 0.1 wt% to 10 wt%, such as from 0.1 wt% to 5 wt%, such as from 0.2 wt% to 1 wt%, such as from 0.1 wt% to 1 wt%, based on the total weight of the filling material.
- 10 **3.** The oral pouched nicotine product according to any one of the preceding claims, wherein the non-tobacco material is a particulate material.
- 4.** The oral pouched nicotine product according to any one of the preceding claims, wherein the non-tobacco material comprises a sugar alcohol such as maltitol and/or cellulose such as microcrystalline cellulose and/or powdered
15 cellulose.
- 5.** The oral pouched nicotine product according to any one of the preceding claims, wherein the non-tobacco material comprises maltitol and/or microcrystalline cellulose.
- 20 **6.** The oral pouched nicotine product according to any one of the preceding claims, wherein the nicotine source comprises one or more of the following: nicotine base, nicotine hydrochloride, nicotine dihydrochloride, nicotine monotartrate, nicotine bitartrate, nicotine bitartrate dihydrate, nicotine sulphate, nicotine zinc chloride monohydrate and nicotine salicylate, nicotine benzoate, nicotine polacrilex.
- 25 **7.** The oral pouched nicotine product according to any one of the preceding claims, wherein the flavouring agent comprises one or more of the following: a synthetic flavour such as a nature-identical flavour, a plant based flavour, a flavour oil, a hydrophobic flavour oil such as an essential oil.
- 8.** The oral pouched nicotine product according to any one of the preceding claims, wherein the flavouring agent
30 comprises one or more of the following: lemon oil, lime oil, orange oil, grapefruit oil, bergamot oil.
- 9.** The oral pouched nicotine product according to any one of the preceding claims, wherein the moisture is present in an amount within the range of from 1 wt% to 20 wt%, such as from 15 wt% to 20 wt%, such as from 12 wt% to 20 wt%, such as from 1 wt% to 12 wt%, such as from 1 wt% to 5 wt%, such as from 0.1 wt% to 1 wt%, based on
35 the total weight of the filling material.
- 10.** The oral pouched nicotine product according to any one of claims 1-8, wherein the moisture is present in an amount within the range of from 20 wt% to 50 wt%, such as from 20 wt% to 45 wt%, such as 40 wt% based on the total weight of the filling material.
- 40 **11.** The oral pouched nicotine product according to any one of the preceding claims, wherein the packaging material comprises or consists of a nonwoven material.
- 12.** The oral pouched nicotine product according to any one of the preceding claims, wherein the acid is present in an amount within the range of from 5 wt% to 15 wt%, 10 wt% to 15 wt% or 5 wt% to 10 wt%, based on the total weight
45 of the packaging material.
- 13.** The oral pouched nicotine product according to any one of the preceding claims, wherein the acid comprises or consists of citric acid.
- 50 **14.** A method for preparing an oral pouched nicotine product according to any one of the preceding claims, the method comprising the steps of:
- a) providing a filling material as defined in any one of the preceding claims, and
55 b) enclosing the filling material in the packaging material obtained in step a).
- 15.** The method according to claim 14, wherein the packaging material is produced by

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(i) treatment of a packaging material with an aqueous solution comprising an acid selected from the group consisting of citric acid, malic acid, lactic acid, ascorbic acid, tartaric acid and any combination thereof, followed by (ii) drying.

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EUROPEAN SEARCH REPORT

Application Number
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