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(54) METHOD OF MANUFACTURING A SMOKELESS ARTICLE

(57) A method of manufacturing a smokeless article is described, along with smokeless articles obtainable by the method. The method includes preparing a precursor blend composition, followed by adding a lubricant selected from stearic acid or a salt thereof to the precursor blend composition and blending the resulting mixture. The smokeless article is useful for oral consumption, e.g. an oral nicotine delivery (OND) article.

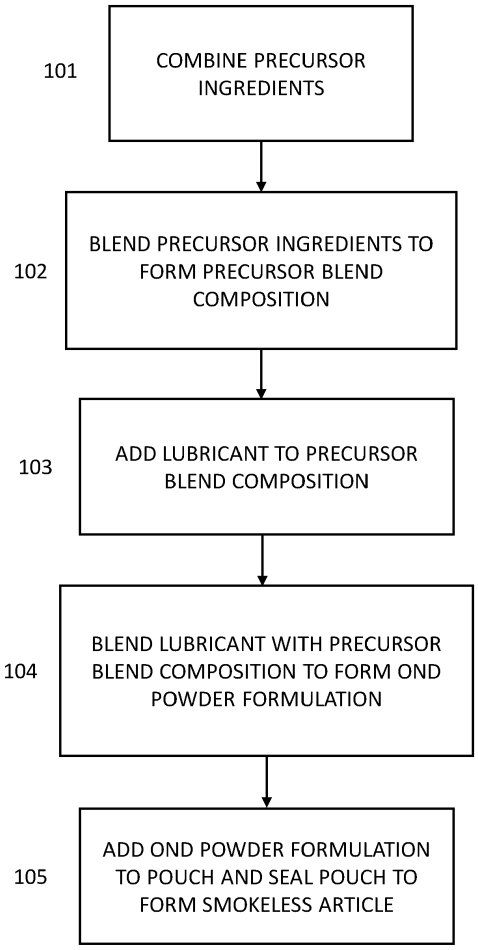

Figure 5

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Description

FIELD

[0001] The present disclosure relates to a method of manufacturing a smokeless article. In particular, the disclosure relates to a method of manufacturing a smokeless article for oral consumption comprising a pouch enclosing an oral nicotine delivery (OND) powder formulation. The disclosure also relates to smokeless articles manufactured using the method.

BACKGROUND

[0002] Smokeless articles are a suitable alternative to conventional cigarettes because they do not require heating for substance delivery to the user. Instead, smokeless articles rely on saliva to extract soluble substances, typically nicotine and/or flavours, from tobacco contained within the smokeless article.

[0003] Smokeless articles are placed in the mouth where saliva extracts the soluble element from the content contained within. Typically, the smokeless article is placed in the oral cavity, sublingually or in the oral vestibule (between the teeth and lips/cheeks). The user may assist extraction by oral manipulation, such as by chewing and/or sucking or pressing on the outside of the mouth to squeeze the pouch.

[0004] The above-described extraction and delivery process continues until the soluble element is depleted from the smokeless article. The smokeless article is then removed from the mouth and disposed of.

[0005] Some commercially available smokeless articles contain snuff. Snuff is smokeless tobacco made from ground or pulverised tobacco leaves. Snuff is available in dry form or wet (moist) form. Moist snuff may be referred to as snus. Two common varieties of snus are Scandinavian snus and American snus. Both varieties of snus are available in a loose form, but are often contained within a saliva permeable pouch.

[0006] There is a need for improved design of smokeless articles to enhance the user experience and improve the function of its constituent components.

[0007] The present disclosure has been devised in the light of the above considerations.

SUMMARY

[0008] At its most general, the present disclosure relates to a method of manufacturing a smokeless article e.g. an oral nicotine delivery (OND) article for oral use.

[0009] The present disclosure provides in a first aspect a method of manufacturing a smokeless article for oral consumption comprising a pouch enclosing a content, e.g. an oral nicotine delivery (OND) powder formulation.

[0010] In some examples, the method comprising the steps of:

(i) preparing a precursor blend composition, followed by

(ii) adding a lubricant selected from stearic acid or a salt thereof to the precursor blend composition and blending the resulting mixture.

[0011] It has been found that the particular combination of (a) the use of stearic acid or a salt thereof as lubricant; and (b) the addition of said lubricant specifically *after* other ingredients of the content have already been mixed to create the precursor blend composition, leads to improved properties of the content. The resultant formulation has a lower Carr's Index (indicating improved compressibility) and a lower Hausner's Ratio (indicating improved flowability) than formulations prepared by other methods or with other types of lubricant.

[0012] The improved flowability and/or compressibility of the content is itself advantageous because it improves the handling of the powder during manufacture.

[0013] In addition, since the powder has improved compressibility and/or flowability, it is possible to increase the water content of the formulation beyond what would normally be possible, while still maintaining acceptable compressibility and flowability. As a result, when a higher water content is used, the user's perception of active ingredient, e.g. nicotine, can be improved, because a higher water content causes the perceived active agent content of the smokeless article to be increased. The end result is a smokeless article which seems "stronger" to the user, which is desirable to many users.

[0014] The method of the invention therefore delivers a smokeless article which is easier to manufacture due to improved compressibility and/or flowability of the constituent content, and which may also deliver improved nicotine perception to the user.

[0015] As used herein, the term "oral consumption" is intended to refer to any oral administration route achieved by placing the smokeless article into the oral cavity. This includes, but is not limited to, buccal, sub-lingual, periodontal,

gingival and ingestion.

[0016] The content may comprise an active agent. The smokeless article may be an oral nicotine delivery (OND) article when the active agent within the content comprises a nicotinic compound.

[0017] In some examples, the active agent comprises or consists of an active compound. In some examples the active agent comprises or consists of a nicotinic compound.

[0018] The smokeless article comprises a pouch enclosing a content, e.g. an OND powder formulation, wherein the content (including e.g. nicotine-dosed non-tobacco plant material fibres) is completely enclosed by the pouch. The pouch is sealed to ensure that the contents of the pouch does not scatter inside the mouth.

[0019] The smokeless article may have a mass of about 0.1 g to 5.0 g, such as about 0.5 g to about 4.0 g or about 1.0 g to about 3.0 g.

[0020] The smokeless article may have a length of about 30 mm, such as about 28 mm or 26 mm, a width of about 12 mm, such as about 10 mm or 8 mm, and a depth of about 5 mm, such as about 4 mm or 3 mm.

[0021] The smokeless article may have an active lifetime of about 20 minutes to about 60 minutes, such as about 25 minutes to 50 minutes or about 30 minutes to about 45 minutes, after being placed in the mouth. As used herein, the term "active lifetime" is intended to refer to the amount of time after being placed in the mouth that the smokeless article provides the user with a perceptible taste and/or physiological experience. For example, for an article containing an active ingredient such as nicotine or other pharmacologically active ingredient the active lifetime may be defined as the in use period of time in which 90%wt of the available pharmacologically active is released. In other words, the active lifetime may be the duration of time from insertion into the oral cavity for 90%wt of the total amount of nicotine pharmacologically active ingredient that is capable of being released during normal use to dissolve into the user's saliva and /or enter the user's bloodstream. It will therefore be appreciated that the active lifetime of a product may vary from user to user and for a user based on oral conditions, in particular extent of salivation. Nonetheless, the skilled person is able to mimic oral conditions to determine the active lifetime in one instance, which can be used as a comparison or analysis point.

[0022] The pouch may be formed from one or more materials. The pouch material may be formed from fiber, paper, cloth and fabric. The pouch material may be formed from one or more polymeric materials. The polymeric material may be selected from one or more of hydroxypropyl cellulose (HPC), hydroxypropyl methylcellulose (HPMC), polyvinyl alcohol (PVOH), polyvinylpyrrolidone (PVP), polyethylene oxide (PEO) hydroxyethyl cellulose (HEC), polyethylene glycol (PEG), pullulan, sodium alginate, xanthan gum, tragancanth gum, guar gum, acacia gum, arabic gum, polyacrylic acid, maltodextrin, methylmethacrylate copolymer, carboxyvinyl copolymers, starch and gelatin.

[0023] The pouch is typically completely insoluble in saliva. Suitable insoluble pouch materials include, but are not limited to, fiber, paper, water-insoluble polymers, cloth and fabric. Suitable soluble pouch materials include, but are not limited to, water-soluble polymers such as polyethylene oxide (PEO), hydroxypropyl cellulose (HPC) and hydroxypropyl methylcellulose (HPMC).

[0024] The pouch may be formed by, for example, folding a single sheet on itself or bringing two or more sheets together and sealing the edges. The edges may initially be partially sealed to provide an open pouch in which the content (e.g. nicotine-dosed non-tobacco plant material fibres) may be placed before completely sealing the pouch closed. The sheets may be the same thickness or different thicknesses.

[0025] The pouch is porous. In some examples, at least 50% of the pores have a diameter of 50 μm to 200 μm, such as 100 μm to 175 μm or 125 μm or 150 μm. In some examples, at least 50% of the pores have a diameter of at least 100 μm. For example, in some examples at least 55%, 60%, 65%, 70%, 75%, 80%, 85%, 90%, 95% or 100% of the pores have such diameters.

[0026] The pouch may be coloured or include markings, such as brand logos and text, to improve user perception. The pouch may be partially or completely coloured by a colourant.

[0027] The lubricant used in the process is selected from stearic acid or a salt thereof. Such compounds provide surprising improvements in powder flow and compressibility which are not observed for alternative lubricants, such as fumarate salts (even those which contain a stearyl group).

[0028] The lubricant may consist of a single compound selected from stearic acid or a salt thereof, or may comprise a mixture of two or more different compounds, at least one of which being selected from selected from stearic acid or a salt thereof. The lubricant may comprise a mixture of two or more different compounds, for example two, three or four different compounds, each of which being selected from selected from stearic acid or a salt thereof.

[0029] In some examples, the lubricant comprises or consists of stearic acid. Stearic acid has been found to be a particularly effective lubricant.

[0030] In some examples, the lubricant comprises or consists of a salt of stearic acid. In some examples, the lubricant comprises or consists of a metal salt of stearic acid.

[0031] In some examples, the lubricant comprises or consists of an alkaline earth metal salt of stearic acid. Alkaline earth metal salts of stearic acid, such as magnesium, calcium or strontium stearate, are a particularly useful category of salt of stearic acid for use as a lubricant.

[0032] In some examples, the lubricant comprises or consists of magnesium stearate. Magnesium stearate has been

found to be a particularly effective lubricant.

[0033] In some examples, the lubricant consists of stearic acid or a salt thereof. In some examples, the lubricant consists of stearic acid or magnesium stearate.

[0034] In some examples, the lubricant (stearic acid or salt thereof) is a particulate solid material. In some examples, the lubricant is a particulate solid material having an average particle size D_v50 of from 5 to 15 μm, for example from 5 to 12 μm or 7 to 11 μm. D_v50 is the value at which half of the particle population in a volume distribution lies below the value and half lies above the value, and it is determined using laser diffraction, for example using a Malvern Mastersizer 3000 under the Mie scattering theory.

[0035] Magnesium stearate is commercially available and one possible source is LIGAMED MF-2-V, supplied by IMCD. This product has an average particle size of 7-11 μm.

[0036] Stearic acid is commercially available and one possible source is LIGAMED SA-1-V, supplied by IMCD.

[0037] The method comprises preparing a precursor blend composition. In some examples, preparing the precursor blend composition comprises mixing together components of the precursor blend composition in high-shear mixing apparatus.

[0038] Suitable mixing apparatus is known to the skilled person. One example of mixing apparatus which may be used for powder blending is a Cube Mixer KB, which can be attached to an Erweka AR403 all purpose system to provide mixing apparatus.

[0039] The method comprises separate steps of (i) preparing a precursor blend composition, followed by (ii) adding the lubricant and blending the resulting mixture.

[0040] In some examples, the precursor blend composition comprises or consists of plant fibres and a humectant. In some examples, the humectant comprises or consists of a polyhydric alcohol and water. In some examples, the humectant comprises or consists of glycerol and water. In some examples, the plant fibres comprise or consist of cellulose fibres, for example in the form of powdered cellulose.

[0041] In some examples, the precursor blend composition does not contain any stearic acid or salts thereof. In some examples, the precursor blend composition does not contain any powdered lubricant materials.

[0042] In some examples, the precursor blend composition does not contain any source of nicotine.

[0043] In some examples, the precursor blend composition comprises plant fibres, a nicotine source, water and flavourant.

[0044] In some examples, the nicotine source comprises a nicotinic compound selected from nicotine, nicotine salt(s), nicotine complex(es); and nicotine solvate(s).

[0045] In some embodiments, the nicotine source is provided in a plant material. In some embodiments, the plant material is tobacco.

[0046] In some embodiments, the smokeless article is tobacco-free.

[0047] In this way, the user may experience a similar or enhanced recreational/pharmaceutical effect as compared to conventional tobacco-containing products without experiencing undesirable components inherent to tobacco (e.g. tobacco flavour).

[0048] In some embodiments, the total nicotine content is from 5 to 15 mg, preferably about 10 mg.

[0049] In some examples, the plant fibres comprise or consist of one or more of wheat fibres, cellulose fibres, bamboo fibres, pine fibres and eucalyptus fibres.

[0050] In some examples, the precursor blend composition further comprises sodium chloride, ammonium chloride, propylene glycol and sodium carbonate.

[0051] Thus in some examples, preparing the precursor blend composition comprises mixing together two or more components selected from plant fibres, a nicotine source, water, flavourant, sodium chloride, ammonium chloride, propylene glycol and sodium carbonate. In some examples, preparing the precursor blend composition comprises mixing together two or more components selected from plant fibres, a nicotine source, water, flavourant, sodium chloride, ammonium chloride, propylene glycol and sodium carbonate in high-shear mixing apparatus.

[0052] In some examples, the precursor blend composition comprises or consists of:

water, in an amount of from 40 wt% to 60 wt%, for example from 50 wt% to 60 wt%, based on the total weight of the content;

glycerol, in an amount of from 5 wt% to 20 wt%, for example from 5 wt% to 10 wt%, based on the total weight of the content; and

plant fibres (e.g. cellulose fibres), in an amount of from 20 wt% to 60 wt%, for example from 35 wt% to 45 wt%, based on the total weight of the content.

[0053] In some examples, the precursor blend composition comprises or consists of:

water, in an amount of from 40 wt% to 60 wt%, for example from 50 wt% to 60 wt%, based on the total weight of the content;

glycerol, in an amount of from 5 wt% to 20 wt%, for example from 5 wt% to 10 wt%, based on the total weight of the content;

a nicotine source, for example a nicotine salt, in an amount of from 0.5 wt% to 5 wt%, for example from 1.0 wt% to 1.0 wt%, based on the total weight of the content; and

plant fibres (e.g. cellulose fibres), in an amount of from 20 wt% to 60 wt%, for example from 35 wt% to 45 wt%, based on the total weight of the content.

[0054] In some examples, step (i) of preparing a precursor blend composition comprises adding each component of the precursor blend composition to a suitable mixer and mixing for a predetermined period of time. In some examples, mixing is continued until a homogeneous mixture is obtained. In some embodiments, step (i) comprises first mixing the liquid components of the precursor blend composition together, before adding the mixed liquid components to the solid components while mixing, until a homogeneous composition is obtained.

[0055] After the precursor blend composition has been prepared, the lubricant is added to the precursor blend and the resulting mixture of precursor blend composition and lubricant is blended.

[0056] The inventors have found that separate addition of the lubricant *after* preparation of the precursor blend composition provides a smokeless article with improved properties relative to a corresponding smokeless article in which the lubricant is added together with all other components at the start.

[0057] In some examples, blending the resulting mixture after addition of the lubricant comprises mixing together the precursor blend composition and the lubricant in mixing apparatus for at least 3 minutes. In some examples mixing is performed for at least 4 minutes, for example about 5 minutes.

[0058] In some examples, blending the resulting mixture after addition of the lubricant comprises mixing together the precursor blend composition and the lubricant in a tumbling mixer.

[0059] In some examples mixing is performed in a mixer operating at 10-20 RPM, for example about 15 RPM.

[0060] In some examples, blending the resulting mixture after addition of the lubricant comprises mixing together the precursor blend composition and the lubricant in a tumbling mixer operating at 10-20 RPM, for example about 15 RPM.

[0061] In some examples, after addition of the lubricant the mixture is blended for 4-6 minutes at 10-20 RPM. In some examples, blending the resulting mixture after addition of the lubricant comprises mixing together the precursor blend composition and the lubricant in a tumbling mixer for 4-6 minutes at 10-20 RPM.

[0062] Such mixing conditions have been found to provide optimal coating of the particles of the precursor blend composition with lubricant, providing a smokeless article with improved properties.

[0063] In some examples, the amount of lubricant in the content is from 1 wt% to 10 wt%, for example from 1 wt% to 5 wt%, based on the total weight of the content. In some examples, the amount of lubricant in the content is from 3 wt% to 5 wt%, based on the total weight of the content.

[0064] In some examples, the amount of water in the content is from 40 wt% to 60 wt%, for example from 45 wt% to 55 wt%, based on the total weight of the OND powder formulation. In some examples, the amount of water in the content is from 50 wt% to 55 wt%, based on the total weight of the content.

[0065] In some examples, the amount of glycerol in the content is from 5 wt% to 20 wt%, for example from 5 wt% to 15 wt%, based on the total weight of the content. In some examples, the amount of glycerol in the content is from 5 wt% to 10 wt%, based on the total weight of the content.

[0066] In some examples, the amount of plant fibres (e.g. cellulose fibres) in the content is from 20 wt% to 60 wt%, for example from 30 wt% to 45 wt%, based on the total weight of the content. In some examples, the amount of plant fibres (e.g. cellulose fibres) in the content is from 35 wt% to 40 wt%, based on the total weight of the content.

[0067] In some examples, the amount of nicotine in the content is from 0.5 wt% to 5 wt%, for example from 0.5 wt% to 2.0 wt%, based on the total weight of the content. In some examples, the amount of nicotine in the content is from 1.2 wt% to 1.6 wt%, based on the total weight of the content.

[0068] In some examples, the content comprises or consists of:

the lubricant in an amount of from 1 wt% to 10 wt%, for example from 1 wt% to 5 wt%, based on the total weight of the content;

water, in an amount of from 40 wt% to 60 wt%, for example from 45 wt% to 55 wt%, based on the total weight of the content;

glycerol, in an amount of from 5 wt% to 20 wt%, for example from 5 wt% to 15 wt%, based on the total weight of the content; and

plant fibres (e.g. cellulose fibres), in an amount of from 20 wt% to 60 wt%, for example from 30 wt% to 45 wt%, based on the total weight of the content.

[0069] In some examples, the content comprises or consists of:

the lubricant in an amount of from 1 wt% to 10 wt%, for example from 1 wt% to 5 wt%, based on the total weight of the content;

water, in an amount of from 40 wt% to 60 wt%, for example from 45 wt% to 55 wt%, based on the total weight of the content;

glycerol, in an amount of from 5 wt% to 20 wt%, for example from 5 wt% to 15 wt%, based on the total weight of the content;

plant fibres (e.g. cellulose fibres), in an amount of from 20 wt% to 60 wt%, for example from 30 wt% to 45 wt%, based on the total weight of the content; and

a nicotine source (e.g. nicotine salt), in an amount of from 0.5 wt% to 5 wt%, for example from 0.5 wt% to 2.0 wt%, based on the total weight of the content.

[0070] The nicotine source may include at least one nicotine salt selected from the group consisting of nicotine hydrochloride, nicotine dihydrochloride, nicotine monotartrate, nicotine bitartrate, nicotine bitartrate dihydrate, nicotine sulfate, nicotine zinc chloride monohydrate, nicotine salicylate and mixtures thereof.

[0071] In some examples, the method further comprises, after the step (ii) of blending the precursor blend composition with the lubricant, adding the content to a suitable pouch and sealing the pouch to form a smokeless article.

[0072] The present disclosure may also provide in a second aspect a smokeless article for oral consumption. The smokeless article may comprise a pouch enclosing an oral nicotine delivery (OND) powder formulation.

[0073] As explained above, the addition of the lubricant at a specific point in the process allows the water content to be increased without detrimentally impacting powder flow or compressibility, thereby improving nicotine perception for the user. The content may comprise a lubricant selected from stearic acid or a salt thereof, and at least 45 wt% water, based on the total weight of the content, for example at least 46 wt%, at least 47 wt%, at least 48 wt%, at least 49 wt%, at least 50 wt%, at least 51 wt% or at least 52 wt%. Such high water content has not been previously possible without detrimental effects on other properties of the formulation such as powder flow and compressibility.

[0074] In some examples, the content (e.g. OND powder formulation) has a Carr's Index of less than 35, for example less than 34, less than 33, less than 32 or less than 31.

[0075] Carr's Index may be determined by calculating both the tapped and bulk density of the content, e.g. using the method in Ph. Eur. 2.9.34. Carr's Index is then given by:

$$Carr's\ Index = 100 - \frac{(Tapped\ Density - Bulk\ Density)}{Tapped\ Density}$$

[0076] In some examples, the content has a Hausner's ratio of less than 1.5, for example less than 1.49, less than 1.48, less than 1.47 or less than 1.46.

[0077] Hausner's ratio may be determined by calculating both the tapped and bulk density of the content, e.g. using the method in Ph. Eur. 2.9.34. Hausner's ratio is then given by:

$$Hausner's\ Ratio = \frac{Tapped\ Density}{Bulk\ Density}$$

[0078] In some examples, the content has a Carr's Index of less than 35, for example less than 34, less than 33, less than 32 or less than 31; and a Hausner's ratio of less than 1.5, for example less than 1.49, less than 1.48, less than 1.47 or less than 1.46. In some examples, the content has a Carr's Index of less than 31; and a Hausner's ratio of less than 1.46.

[0079] In some examples, the content has a bulk density above about 0.35 g/mL, for example above about 0.38 g/mL, above about 0.40 g/mL, or above about 0.41 g/mL.

[0080] In some examples, the content has a bulk density up to about 0.50 g/mL, for example up to about 0.45 g/mL, up to about 0.44 g/mL, up to about 0.43 g/mL, or up to about 0.425 g/mL.

[0081] In some examples the content has a bulk density of 0.35-0.50 g/mL, 0.38-0.45 g/mL, 0.40-0.45 g/mL, 0.41-0.44 g/mL, 0.41-0.43 g/mL, or 0.411-0.421 g/mL.

[0082] In some examples, the content has a tapped density above about 0.55 g/mL, for example above about 0.57 g/mL, above about 0.58 g/mL, or above about 0.59 g/mL.

[0083] In some examples, the content has a tapped density up to about 0.65 g/mL, for example up to about 0.64 g/mL, up to about 0.62 g/mL, up to about 0.60 g/mL, or up to about 0.595 g/mL.

[0084] In some examples the content has a tapped density of 0.55-0.65 g/mL, 0.58-0.62 g/mL, 0.58-0.60 g/mL, 0.59-0.60 g/mL, 0.59-0.595 g/mL, or 0.591-0.594 g/mL.

[0085] In some examples, the content has a Carr's Index of less than 31, a Hausner's ratio of less than 1.46 and a bulk density of 0.35-0.50 g/mL. In some examples, the content has a Carr's Index of less than 31, a Hausner's ratio of less than 1.46, a bulk density of 0.35-0.50 g/mL and a tapped density of 0.55-0.65 g/mL.

[0086] Tapped density can be measured using the method described in the Examples.

[0087] In some examples, the content comprises plant fibres, a nicotine source, water and flavourant.

[0088] In some examples, the smokeless article is made by a method according to the first aspect described above. Thus in some examples the smokeless article is made by a method comprising the steps of:

(i) preparing a precursor blend composition, followed by

(ii) adding a lubricant selected from stearic acid or a salt thereof to the precursor blend composition and blending the resulting mixture.

[0089] The content (also referred to herein as "contents" or "pouch contents") may comprise nicotine-dosed plant material fibres and may comprise one or more additional substances. The contents may comprise humectant, plant material fibres and the lubricant, and may comprise one or more additional substances. The contents may consist of humectant, plant material fibres and the lubricant.

[0090] The or each additional substance may individually be a biologically/pharmacologically active compound, pH stabilisers or adjusters, humectants, flavourants, fillers, preservatives, aqueous/non-aqueous solvents and binders. The or each additional substance may be provided for more than one purpose.

[0091] The contents of the pouch (i.e. the ingredients, material and/or substances enclosed within the pouch) preferably occupies substantially all of the internal volume of the pouch. The contents may occupy 80%, 85%, 90%, 95% or 100% of the internal volume of the pouch. The contents may comprise a solid material to provide physical integrity, such as an organic material (e.g. plant material) or an inorganic material. Such solid materials may naturally or inherently contain one or more biologically/pharmacologically active compounds and/or additives.

[0092] **Biologically/pharmacologically active compounds** are provided to produce a pharmacological effect in the user. Suitable biologically/pharmacologically active compounds include the group consisting of: nicotine, cocaine, caffeine, opiates and opioids, cathine and cathinone, kavalactones, mysticin, beta-carboline alkaloids, salvinorin A together with any combinations, functional equivalents to, and/or synthetic alternatives of the foregoing. Biologically/-pharmacologically active compounds may also have additive properties.

[0093] In some embodiments the content includes an active compound comprising nicotine and wherein the form of nicotine is selected from the group consisting of nicotine salts, nicotine base, stabilized nicotine and mixtures thereof. For example, the content may include at least one nicotine salt selected from the group consisting of nicotine hydrochloride, nicotine dihydrochloride, nicotine monotartrate, nicotine bitartrate, nicotine bitartrate dihydrate, nicotine sulfate, nicotine zinc chloride monohydrate, nicotine salicylate and mixtures thereof.

[0094] **pH stabilisers or adjusters** may be provided to adjust the user experience and/or modify the bioavailability of a pharmacologically active compound. For instance, under acidic conditions, nicotine is protonated and does not readily cross mucous membranes. Examples of suitable pH stabilisers include ammonia, ammonium carbonate, sodium carbonate and calcium carbonate. The overall pH of the smokeless article is preferably pH 7 to pH 9, such as pH 7.25 to pH 8.75 or pH 7.5 to pH 8.5.

[0095] The overall pH of a smokeless article may be determined by, for example, (i) placing the smokeless article in 10 mL of distilled water (ii) agitating the mixture for at least 5 minutes and (iii) measuring the pH of the solution with a pH probe.

[0096] **Fillers** may be provided to increase the volume of the smokeless article (e.g. by increasing the volume contained within the pouch and to strengthen the contents). Suitable fillers include calcium carbonate, calcium phosphate, corn starch, grains, lactose, polysaccharides (e.g. maltodextrin), polyols, sugars (e.g. dextrose, manitol, xylitol, sorbitol), natural fibres (e.g. non-tobacco fibres), microcrystalline cellulose, cellulose and cellulose derivatives (e.g. finely divided cellulose), lignocellulose fibres (e.g. wood fibres), jute fibres and combinations thereof. In some cases, the filler content is 5 to 10 wt% of the contents e.g. around 6 to 9 wt%.

[0097] **Flavourants** may be provided in solid or liquid form. Suitable flavourants include coffee, eucalyptus, menthol, liquorice, peppermint, spearmint, chocolate, fruit flavour (including e.g. citrus, cherry etc.), vanilla, spice (e.g. ginger, cinnamon) and tobacco flavour. The flavourant may be evenly dispersed throughout the contents or may be provided in isolated locations and/or varying concentrations throughout the contents. As used herein, the term "flavourant" denotes a compound having a desirable taste, aroma or both.

[0098] **Humectants** may be provided to control moisture content thereby preventing the smokeless article from drying out during storage and reducing the amount of saliva wetting required before the user experience begins. Suitable humectants include polyhydric alcohols (e.g. propylene glycol (PG), triethylene glycol, 1 ,2-butane diol and glycerol such as vegetable glycerine (VG)) and their esters (e.g. glycerol mono-, dior tri-acetate). The humectant component may consist of a single humectant compound or may consist of two or more different humectant compounds.

[0099] The humectant may have a lower limit of at least 1 % by weight of the contents such as at least 2 wt%, such as at least 5 wt%, such as at least 10 wt%, such as at least 20 wt%, such as at least 30 wt%, or such as least 40 wt%.

[0100] The humectant may have an upper limit of at most 70% by weight of the contents, such as at most 65 wt%, such as at most 60 wt%, or such as at most 20 wt%, such as at most 10 wt %, such as at most 5 wt %, such as at most 2 wt%.

[0101] Preferably, the amount of humectant is 1 to 70 wt% of the contents, such as 10 to 60 wt% or 50 to 60 wt%.

[0102] Preferably, the contents has an overall amount of water of between 5 and 60 wt% based on the weight of the contents such as between 40 to 60 wt% or 50 to 60 wt%, for example 52 to 60 wt%.

[0103] Smokeless articles having a total moisture content of 10% or less are generally considered to be 'dry'. Smokeless articles having a total moisture content of 40% or more are generally considered to be 'wet'.

[0104] The humectant in the OND powder formulation may comprise or consist of water and one or more polyhydric alcohols, for example water and glycerol.

[0105] **Sweeteners** may be provided to modify the user taste perception and, in particular, overcome bitter flavours that result from other substances. Suitable sweeteners include honey, sugar, brown sugar, glucose, fructose, sucrose, aspartame, xylitol, maltitol, saccharin sodium, glycyrrhizin tripotassium liquorice, jujube or a mixture thereof. The amount of sweetener is in some cases 1 to 20 % by weight of the contents, such as 2 to 15 wt% or 5 to 10 wt%.

[0106] **Stabilisers** are provided to prevent decomposition or degradation over time during storage by, for example, retarding oxidation or unwanted biological activity. Stabilisers may be selected from the group consisting of antioxidants including vitamin E, such as tocopherole, ascorbic acid, sodium pyrosulfite, butylhydroxytoluene, butylated hydroxyanisole, edetic acid and salts thereof; and preservatives including citric acid, tartaric acid, lactic acid, malic acid, acetic acid, benzoic acid, sorbic acid and salts thereof.

[0107] **Binders** may be provided. Suitable binders include starches and/or cellulosic binders such as methyl cellulose, ethyl cellulose, hydroxypropyl cellulose, hydroxyethyl cellulose and carboxymethyl cellulose, gums such as xanthan, guar, arabic and/or locust bean gum, organic acids and their salts such as alginic acid (sodium alginate), agar and pectins. In some embodiments the binder content is 5 to 10 wt% of the contents e.g. around 6 to 9 wt% or 7 to 8 wt%.

[0108] **Colourants** may be provided to modify the user impression of the smokeless article. Colourants include whitening agents. Colourants may be selected from one or more of common colourants such as curcumin (E100), turmeric (E100(ii)), riboflavin (E101), riboflavin-5'-phosphate (E101(ii)), tartrazine (E102), quinoline yellow (E104), riboflavin-5-sodium phosphate (E106), yellow 2G (E107), sunset yellow FCF (E110), carmine, cochineal (E120), azorubine (E122), amaranth (E123), ponceau 4R (E124), erythrosine (E127), red 2G (E128), allura red AC (E129), patent blue V (E131), indigotine (E132), brilliant blue FCF (E133), chlorophylls (E140), copper complexes of chlorophyll (E141), green S (E142), caramel (E150a-d), brilliant black BN (E151), carbon (E153), brown FK (E154), brown HT (E155), alfa-, beta- and gamma- carotene (E160a), annatto, bixin, norbixin (E160b), bell pepper (Paprika) extract (E160c), lycopene (E160d), beta-apo-8'-carotenal (E160e), ethyl ester of beta-apo-8'-carotenic acid (E160f), flavoxanthin (E161a), lutein (E161b), cryptoxanthin (E161c), rubixanthin (E161d), violaxanthin (E161e), rhodoxanthin (E161f), canthaxanthin (E161g), citranaxanthin (E161h), beetroot extract (E162), anthocyanins (E163), calcium carbonate (E170), titanium dioxide (E171), iron oxides (E172), aluminium (E173), silver (E174), gold (E175), lithol rubine BK (E180), tannins (E181). The amount of colourant may be up to about 3% by weight of the smokeless article, such as about 0.5% to about 2.5% or about 1% to about 2%.

[0109] **Plant material** may be provided for physical integrity and may function as a natural source of substances such as, for example, biologically/pharmacologically active compounds, flavourants, pH stabilisers etc. The plant material may comprise least one plant material selected from the list including *Amaranthus dubius, Arctostaphylos uva-ursi* (Bearberry), *Argemone mexicana, Amica, Artemisia vulgaris,* Yellow Tees, *Galea zacatechichi, Canavalia maritima* (Baybean), *Cecropia mexicana* (Guamura), *Cestrum noctumum, Cynoglossum virginianum* (wild comfrey), *Cytisus scoparius, Damiana, Entada rheedii, Eschscholzia califomica* (California Poppy), *Fittonia albivenis, Hippobroma longiflora, Humulus japonica* (Japanese Hops), *Humulus lupulus* (Hops), *Lactuca virosa* (Lettuce Opium), *Laggera alata, Leonotis leonurus, Leonurus cardiaca* (Motherwort), *Leonurus sibiricus* (Honeyweed), *Lobelia cardinalis, Lobelia inflata* (Indian-tobacco), *Lobelia siphilitica, Nepeta cataria* (Catnip), *Nicotiana species* (Tobacco), *Nymphaea alba* (White Lily), *Nymphaea caerulea* (Blue Lily), Opium poppy, *Passiflora incamata* (Passionflower), *Pedicularis densiflora* (Indian Warrior), *Pedi-*

cularis groenlandica (Elephant's Head), *Salvia divinorum, Salvia dorrii* (Tobacco Sage), Salvia species (Sage), *Scutellaria galericulata, Scutellaria lateriflora, Scutellaria nana, Scutellaria* species (Skullcap), *Sida acuta* (Wireweed), *Sida rhombifolia, Silene capensis, Syzygium aromaticum* (Clove), *Tagetes lucida* (Mexican Tarragon), *Tarchonanthus camphoratus, Tumera diffusa* (Damiana), *Verbascum* (Mullein), *Zamia latifolia* (Maconha Brava) together with any combinations, functional equivalents to, and/or synthetic alternatives of the foregoing.

[0110] The plant material may be tobacco. Any type of tobacco may be used. This includes, but is not limited to, flue-cured tobacco, burley tobacco, Maryland Tobacco, dark-air cured tobacco, oriental tobacco, dark-fired tobacco, perique tobacco and rustica tobacco. This also includes blends of the above mentioned tobaccos.

[0111] Any suitable parts of the tobacco plant may be used. This includes leaves, stems, roots, bark, seeds and flowers.

[0112] The tobacco may comprise one or more of leaf tobacco, stem tobacco, tobacco powder, tobacco dust, tobacco derivatives, expanded tobacco, homogenised tobacco, shredded tobacco, extruded tobacco, cut rag tobacco and/or reconstituted tobacco (e.g. slurry recon or paper recon).

[0113] The contents may comprise at least 30 wt% plant material based on the weight of the contents, e.g. at least 35 wt% plant material e.g. around 37 wt% plant material. The contents may comprise 80 wt% or less plant material e.g. 75 wt% or less, or 70 wt% or less plant material.

[0114] The contents may comprise a gathered sheet of homogenised (e.g. paper/slurry recon) tobacco or gathered shreds/strips formed from such a sheet.

[0115] The sheet may have a grammage greater than or equal to 100 g/m^2, e.g. greater than or equal to 110 g/m^2 such as greater than or equal to 120 g/m^2. The sheet may have a grammage of less than or equal to 300 g/m^2 e.g. less than or equal to 250 g/m^2 or less than or equal to 200 g/m^2. The sheet may have a grammage of between 120 and 190 g/m^2.

[0116] The present disclosure may provide a use of the smokeless article according to the second aspect for oral delivery of nicotine to a user.

[0117] A third aspect of the invention is a method of improving the powder flowability and/or powder compressibility of the powder content of a smokeless article, the method comprising:

(i) preparing a precursor blend composition, followed by

(ii) adding a lubricant selected from stearic acid or a salt thereof to the precursor blend composition and blending the resulting mixture.

[0118] In some examples, powder compressibility is characterised by the Carr's Index of the powder and powder flowability is characterised by the Hausner's ratio of the powder.

[0119] In some examples, improving the powder flowability comprises reducing the Hausner's ratio of the powder. In some examples, improving the powder compressibility comprises reducing the Carr's Index of the powder.

[0120] A fourth aspect of the invention is a method of improving the nicotine perception of a smokeless article, the method comprising:

(i) preparing a precursor blend composition, followed by

(ii) adding a lubricant selected from stearic acid or a salt thereof to the precursor blend composition and blending the resulting mixture.

[0121] All of the options and preferences set out above with respect to the method of the first aspect apply equally to the methods of the third and fourth aspects.

[0122] A fifth aspect is a method of manufacturing a smokeless article for oral consumption comprising a pouch enclosing a content, the method comprising the steps of:

(i) preparing a precursor blend composition, followed by

(ii) adding a lubricant to the precursor blend composition and blending the resulting mixture to provide the content;

wherein the content satisfies one or more of the following:

(a) a Carr's Index of less than 31;

(b) a Hausner's ratio of less than 1.46;

(c) a bulk density of 0.35-0.50 g/mL;

(d) a tapped density of 0.55-0.65 g/mL;

(e) an amount of lubricant from 1 wt% to 10 wt%, for example from 1 wt% to 5 wt%, based on the total weight of the content.

[0123] In some examples, the content has a bulk density above about 0.35 g/mL, for example above about 0.38 g/mL, above about 0.40 g/mL, or above about 0.41 g/mL.

[0124] In some examples, the content has a bulk density up to about 0.50 g/mL, for example up to about 0.45 g/mL, up to about 0.44 g/mL, up to about 0.43 g/mL, or up to about 0.425 g/mL.

[0125] In some examples the content has a bulk density of 0.35-0.50 g/mL, 0.38-0.45 g/mL, 0.40-0.45 g/mL, 0.41-0.44 g/mL, 0.41-0.43 g/mL, or 0.411-0.421 g/mL.

[0126] In some examples, the content has a tapped density above about 0.55 g/mL, for example above about 0.57 g/mL, above about 0.58 g/mL, or above about 0.59 g/mL.

[0127] In some examples, the content has a tapped density up to about 0.65 g/mL, for example up to about 0.64 g/mL, up to about 0.62 g/mL, up to about 0.60 g/mL, or up to about 0.595 g/mL.

[0128] In some examples the content has a tapped density of 0.55-0.65 g/mL, 0.58-0.62 g/mL, 0.58-0.60 g/mL, 0.59-0.60 g/mL, 0.59-0.595 g/mL, or 0.591-0.594 g/mL.

[0129] In some examples, the content has a Carr's Index of less than 31, a Hausner's ratio of less than 1.46 and a bulk density of 0.35-0.50 g/mL. In some examples, the content has a Carr's Index of less than 31, a Hausner's ratio of less than 1.46, a bulk density of 0.35-0.50 g/mL and a tapped density of 0.55-0.65 g/mL.

[0130] Tapped density can be measured using the method described in the Examples.

[0131] All the options and preferences set out above in relation to the method of the fist aspect apply equally to the method of the fifth aspect, but for brevity are not repeated here.

[0132] The skilled person will appreciate that except where mutually exclusive, a feature or parameter described in relation to any one of the above aspects may be applied to any other aspect. Furthermore, except where mutually exclusive, any feature or parameter described herein may be applied to any aspect and/or combined with any other feature or parameter described herein.

[0133] The preceding summary is provided for purposes of summarizing some examples to provide a basic understanding of aspects of the subject matter described herein. Accordingly, the above-described features should not be construed to narrow the scope or spirit of the subject matter described herein in any way. Moreover, the above and/or proceeding examples may be combined in any suitable combination to provide further examples, except where such a combination is clearly impermissible or expressly avoided. Other features, aspects, and advantages of the subject matter described herein will become apparent from the following text and the accompanying drawings.

BRIEF DESCRIPTION OF THE FIGURES

[0134] Aspects, features and advantages of the present disclosure will become apparent from the following description of examples in reference to the appended drawings in which like numerals denote like elements.

Figure 1 shows a cross sectional view of a first embodiment of a smokeless article.

Figure 2 shows a cross sectional view of a second embodiment of a smokeless article.

Figure 3 shows a cross sectional view of a third embodiment of a smokeless article.

Figure 4 shows a cross sectional view of a fourth embodiment of a smokeless article.

Figure 5 shows a flow diagram representing a manufacturing method according to the invention.

DETAILED DESCRIPTION OF EMBODIMENTS

[0135] As shown in Figure 1 there is provided a first embodiment of a smokeless article 10 having a pouch 12 containing contents (OND powder formulation) 14. The pouch 12 is substantially rectangular. The pouch 12 is formed from a single sheet of material and is substantially filled by the contents 14. The pouch 12 has a seal 16 along each of the three edges where the inner face of the single sheet meets itself to seal the contents 14 in the pouch 12.

[0136] Figure 2 shows a second embodiment of a smokeless article 10' having a pouch 12 containing contents (OND powder formulation) 14. The pouch 12 is substantially circular. The pouch 12 is formed from two opposing sheets of material and is substantially filled by the contents 14. The pouch has a circumferential seal 16 along the edges where the

two opposing sheets of material meet to seal the contents 14 in the pouch 12.

[0137] Figure 3 shows a third embodiment of a smokeless article 10" that, like the first embodiment, has a pouch 12 made from a single sheet of material. However, one of the three seals 16' is formed by an overlap of the inner face and the outer face of the single sheet meet to seal the contents (OND powder formulation) 14 in the pouch 12. The remaining two seals at opposing ends of the pouch 12 are formed where the inner face of the single sheet meets itself.

[0138] Figure 4 shows a fourth embodiment of a smokeless article 10‴ that comprises the third embodiment enclosed by outer pouch 12" having an outer contents 14" positioned in the space between the inner pouch 12' and the outer pouch 12". The outer pouch 12" also has a circumferential seal 16‴ to seal the outer contents 14" and inner pouch 12' in the outer pouch 12".

[0139] Use of the fourth embodiment begins when the smokeless article 10‴ is placed in the user's mouth where it is exposed to saliva. Saliva first permeates outer pouch 12" and dissolves and extracts the saliva soluble substances of outer contents 14". Upon leaving the outer pouch 12", the saliva soluble substances of outer contents 14" therefore provide the user with a first experience. Saliva subsequently further permeates the inner pouch 12' where it dissolves and extracts the saliva soluble substances of inner contents 14'. The saliva soluble substances of inner contents 14' therefore provide the user with a complimentary and secondary experience. When the extractable amount of saliva soluble substances in the inner contents 14' and outer contents 14" drops below perceivable levels the active lifetime of the smokeless article 10‴ has ended.

[0140] Figure 5 details a method of manufacturing a smokeless article. At step 101, all precursor ingredients (i.e. all ingredients of the OND powder composition with the exception of the lubricant) are combined. This may be directly within the mixing vessel of mixing apparatus, or they may be combined outside mixing apparatus before being transferred into the mixing vessel.

[0141] At step 102, the precursor ingredients (i.e. all ingredients of the OND powder composition with the exception of the lubricant) are blended together, for example within suitable mixing apparatus. The precursor ingredients are blended until a precursor blend composition comprising a homogeneous mixture of precursor ingredients is formed.

[0142] At step 103, a lubricant is added to the precursor blend composition. The lubricant is selected from stearic acid or a salt thereof, for example stearic acid or magnesium stearate. The lubricant is added in powder form.

[0143] At step 104, after the addition of lubricant in step 103, the lubricant and the precursor blend composition are blended together, for example within suitable mixing apparatus. The lubricant and the precursor blend composition are blended until an OND powder formulation comprising a homogeneous mixture of ingredients is formed.

[0144] At step 105, the OND powder formulation is added to a suitable pouch and the pouch is sealed, to form a smokeless article for oral delivery of nicotine to a user.

[0145] Before describing several examples implementing the present disclosure, it is to be understood that the present disclosure is not limited by specific construction details or process steps set forth in the following description and accompanying drawings. Rather, it will be apparent to those skilled in the art having the benefit of the present disclosure that the systems, apparatuses and/or methods described herein could be embodied differently and/or be practiced or carried out in various alternative ways.

[0146] Unless otherwise defined herein, scientific and technical terms used in connection with the presently disclosed inventive concept(s) shall have the meanings that are commonly understood by those of ordinary skill in the art, and known techniques and procedures may be performed according to conventional methods well known in the art and as described in various general and more specific references that may be cited and discussed in the present specification.

[0147] Any patents, published patent applications, and non-patent publications mentioned in the specification are hereby incorporated by reference in their entirety.

[0148] All examples implementing the present disclosure can be made and executed without undue experimentation in light of the present disclosure. While particular examples have been described, it will be apparent to those of skill in the art that variations may be applied to the systems, apparatus, and/or methods and in the steps or in the sequence of steps of the methods described herein without departing from the concept, spirit, and scope of the inventive concept(s). All such similar substitutions and modifications apparent to those skilled in the art are deemed to be within the spirit, scope, and concept of the inventive concept(s) as defined by the appended claims.

[0149] The use of the term "a" or "an" in the claims and/or the specification may mean "one," as well as "one or more," "at least one," and "one or more than one." As such, the terms "a," "an," and "the," as well as all singular terms, include plural referents unless the context clearly indicates otherwise. Likewise, plural terms shall include the singular unless otherwise required by context.

[0150] The use of the term "or" in the present disclosure (including the claims) is used to mean an inclusive "and/or" unless explicitly indicated to refer to alternatives only or unless the alternatives are mutually exclusive. For example, a condition "A or B" is satisfied by any of the following: A is true (or present) and B is false (or not present), A is false (or not present) and B is true (or present), and both A and B are true (or present).

[0151] As used in this specification and claim(s), the words "comprising, "having," "including," or "containing" (and any forms thereof, such as "comprise" and "comprises," "have" and "has," "includes" and "include," or "contains" and "contain,"

respectively) are inclusive or open-ended and do not exclude additional, unrecited elements or method steps.

[0152] Unless otherwise explicitly stated as incompatible, or the physics or otherwise of the embodiments, examples, or claims prevent such a combination, the features of examples disclosed herein, and of the claims, may be integrated together in any suitable arrangement, especially ones where there is a beneficial effect in doing so. This is not limited to only any specified benefit, and instead may arise from an "ex post facto" benefit. This is to say that the combination of features is not limited by the described forms, particularly the form (e.g. numbering) of example(s), embodiment(s), or dependency of claim(s). Moreover, this also applies to the phrase "in one embodiment," "according to an embodiment," and the like, which are merely a stylistic form of wording and are not to be construed as limiting the following features to a separate embodiment to all other instances of the same or similar wording. This is to say, a reference to 'an,' 'one,' or 'some' embodiment(s) may be a reference to any one or more, and/or all embodiments, or combination(s) thereof, disclosed. Also, similarly, the reference to "the" embodiment may not be limited to the immediately preceding embodiment. Further, all references to one or more embodiments or examples are to be construed as non-limiting to the claims.

EXAMPLES

Comparative Example 1 (control)

[0153] A powder formulation was prepared containing the following ingredients:

Ingredient	*Amount (g)*	*wt%*
Glycerol	9.45	6.3
Water	82.5	55
Arbocel 300	58.05	38.7

[0154] Arbocel 300 is powdered cellulose supplied by Pharma Excipients.

[0155] This powder formulation was prepared using the following method (Protocol A):

- All the ingredients were dispensed into appropriately sized beakers using a 2 d.p. balance to determine the weights
- The glycerol was then added into the beaker which contained water dispensed in the previous step. This glycerol/-water mixture was then transferred onto a magnetic stirrer with stirrer bar and mixed for approx. 5 mins until the solution appeared homogeneous.
- While the above solution was mixing, the Arbocel 300 was added to a Kenwood food processor, this then was mixed for ~1 min at full speed.
- The solution prepared in step 2 was slowly added to the Kenwood food processor over the course of ~3-4mins while still mixing. After this the Kenwood was stopped, opened and the sides and bottom of the bowl were scraped.
- The Kenwood was then restarted at full power and mixed for ~1-2mins. The Kenwood was shaken while this was happening to ensure complete mixing.
- Finally, the blend was transferred to a Foil pouch for storage.

Example 1

[0156] The following method (Protocol B) was followed to form an OND powder formulation:

- The powder formulation of Comparatiuve Example 1 manufactured using Protocol A was added to a Cube Mixer KB, which was attached to an Erweka AR403 all purpose system.
- Lubricant (magnesium stearate, LIGAMED MF-2-V, supplied by IMCD) was added to the powder formulation
- The mixture was mixed for approx. 5 mins at 15 rpm
- The final blend was transferred to a foil pouch for storage.

[0157] The final OND powder formulation contained the following ingredients:

Ingredient	*Amount (g)*	*wt%*
Glycerol	2.39	5.98
Water	20.9	52.3

(continued)

Ingredient	*Amount (g)*	*wt%*
Arbocel 300	14.71	36.8
Mg stearate	2	5

Example 2

[0158] Protocol B as set out in Example 1 was followed to form an OND powder formulation, except that stearic acid (LIGAMED SA-1-V, supplied by IMCD) was used instead of magnesium stearate.

[0159] The final OND powder formulation contained the following ingredients:

Ingredient	*Amount (g)*	*wt%*
Glycerol	2.44	6.1
Water	21.34	53.35
Arbocel 300	15.02	37.55
Stearic acid	1.2	3

Comparative Example 2

[0160] An OND powder formulation was prepared using Protocol A set out under Comparative Example 1, except that magnesium stearate (LIGAMED MF-2-V, supplied by IMCD) was also added alongside the Arbocel 300 in the third step.

[0161] The final OND powder formulation contained the following ingredients:

Ingredient	*Amount (g)*	*wt%*
Glycerol	9.45	6.3
Water	82.5	55
Arbocel 300	50.55	33.7
Mg stearate	7.5	5

Comparative Example 3

[0162] An OND powder formulation was prepared using Protocol A set out under Comparative Example 1, except that stearic acid (LIGAMED SA-1-V, supplied by IMCD) was also added alongside the Arbocel 300 in the third step.

[0163] The final OND powder formulation contained the following ingredients:

Ingredient	*Amount (g)*	*wt%*
Glycerol	9.45	6.3
Water	82.5	55
Arbocel 300	53.55	35.7
Stearic acid	4.5	3

Comparative Example 4

[0164] Protocol B as set out in Example 1 was followed to form an OND powder formulation, except that sodium stearyl fumarate (PRUV, supplied by Pharma Excipients) was used instead of magnesium stearate. Sodium stearyl fumarate is a compound which is known as a lubricant in pharmaceutical tablet compositions. Sodium stearyl fumarate is a fumarate salt which contains within it a stearyl (1-octadecane) group - it is not a stearate salt.

[0165] The final OND powder formulation contained the following ingredients:

Ingredient	*Amount (g)*	*wt%*
Glycerol	2.47	6.18
Water	21.56	53.9
Arbocel 300	15.17	37.9
Sodium stearyl fumarate	0.8	2

Comparative Example 5

[0166] An OND powder formulation was prepared using Protocol A set out under Comparative Example 1, except that sodium stearyl fumarate (PRUV, supplied by Pharma Excipients) was also added alongside the Arbocel 300 in the third step.

[0167] The final OND powder formulation contained the following ingredients:

Ingredient	*Amount (g)*	*wt%*
Glycerol	9.45	6.3
Water	82.5	55
Arbocel 300	55.05	36.7
Sodium stearyl fumarate	3	2

Example 3

[0168] Carr's Index, Hausner's Ratio, bulk density and tapped density were determined for each of Examples 1-2 and Comparative Examples 1-5, according to the following method based on Ph. Eur. 2.9.34.

1. A 100 mL Cylinder was placed on to a 2 d.p. balance, and tared.

2. Using a beaker, test powder was transferred into the 100 mL cylinder at approx. 90-degree angle until the cylinder was filled to approx. 90-100mL.

3. The volume (V0) was recorded, before tapping. The cylinder was then gently placed back on to the balance and the net weight of the contents was then recorded. The density calculated using V0 and the net weight is the bulk density.

4. The cylinder was then placed on to a JV1000 density tester, and tapped 10 times, after which the volume was recorded as (V10).

5. The cylinder was tapped another 490 times, and once again the volume was recorded as (V500).

6. This was repeated, but the cylinder was tapped a further 750 times and the volume recorded once again as (V1250).

7. If the volume of the powder in the cylinder dropped by more than 1 cm, then the cylinder was tapped a further 1250 times, this was then repeated until the volume in the cylinder dropped by no more than 1cm.

8. This whole procedure was repeated, and averages of the bulk and tapped densities were calculated.

9. The results of the above were used to calculate the Carr's index and Hasuners Ratio as per the calculations below. The lower these numbers are the better the flow of the blends is.

$$Hausner's\ Ratio = \frac{Tapped\ Density}{Bulk\ Density}$$

$$Carr's\ Index = 100 - \frac{(Tapped\ Density - Bulk\ Density)}{Tapped\ Density}$$

[0169] The results are provided in the Table below:

Example	*Bulk Density / g ml^{-1}*	*Tapped Density / g ml^{-1}*	*Carr's Index*	*Hausner's Ratio*
Example 1	0.411	0.594	30.8	1.45
Example 2	0.421	0.591	28.8	1.40
Comparative Example 1	0.366	0.555	34.0	1.52
Comparative Example 2	0.377	0.605	37.8	1.61
Comparative Example 3	0.375	0.581	35.4	1.55
Comparative Example 4	0.370	0.593	37.6	1.60
Comparative Example 5	0.384	0.608	36.8	1.58

[0170] The results show that, when stearic acid or a salt thereof is used as a lubricant, the powder properties (Carr's Index and Hausner's ratio) are improved when the lubricant is added only after the remaining ingredients have already been mixed to form a precursor blend composition. The same improvement is not seen when the lubricant is added at the start of the manufacturing method, alongside all other ingredients before any mixing step. In fact, when the magnesium stearate or stearate salt lubricant is added at this early stage (Comparative Example 2 and Comparative Example 3 respectively), both the Carr's Index and Hausner's Ratio are *increased* (poor compressibility and flowability) relative to a control composition containing no lubricant (Comparative Example 1).

[0171] Furthermore, the improvement observed by adding the lubricant after an initial mixing stage relative to adding the lubricant at the start before any mixing is not observed for all types of lubricant. The results show this improvement when stearic acid or a salt thereof is used as lubricant. However when sodium stearyl fumarate is used as lubricant, both the Carr's Index and Hausner's Ratio are *increased* (poor compressibility and flowability) when the sodium stearyl fumarate is added after an initial precursor mixing stage.

[0172] Thus, improved OND powder formulation properties are observed when a lubricant containing stearic acid or a salt thereof is added to the composition after an initial precursor mixing stage.

[0173] The tests were conducted on samples without any nicotine content, however the results would extend to corresponding samples containing a nicotine source because the observed improvements in physical properties such as powder flowability and compressibility would not be impacted by the presence of the typical small amount of a nicotine source.

Claims

1. A method of manufacturing a smokeless article for oral consumption comprising a pouch enclosing a content, the method comprising the steps of:

   (i) preparing a precursor blend composition, followed by
   (ii) adding a lubricant selected from stearic acid or a salt thereof to the precursor blend composition and blending the resulting mixture.

2. A method according to claim 1, wherein the lubricant comprises or consists of stearic acid.

3. A method according to claim 1 or 2, wherein the lubricant comprises or consists of an alkaline earth metal salt of stearic acid, optionally wherein the lubricant comprises or consists of magnesium stearate.

4. A method according to any one of the preceding claims, wherein the precursor blend composition comprises plant fibres and humectant, optionally wherein the humectant comprises or consists of water and a polyhydric alcohol; optionally wherein the plant fibres comprise or consist of one or more of wheat fibres, cellulose fibres, bamboo fibres, pine fibres and eucalyptus fibres.

5. A method according to claim 4, wherein the precursor blend composition further comprises one or more of sodium chloride, ammonium chloride, propylene glycol and sodium carbonate.

6. A method according to claim 4 or 5, wherein the precursor blend composition further comprises a nicotine source.

7. A method according to any one of the preceding claims, wherein preparing the precursor blend composition comprises mixing together components of the precursor blend composition in mixing apparatus.

8. A method according to any one of the preceding claims, wherein blending the resulting mixture after addition of the lubricant comprises mixing together the precursor blend composition and the lubricant in mixing apparatus for at least 3 minutes.

9. A method according to any of the preceding claims, wherein after addition of the lubricant the mixture is blended for 4-6 minutes at 10-20 RPM.

10. A method according to any of the preceding claims, wherein the amount of lubricant in the OND powder formulation is from 1 wt% to 10 wt%, for example from 1 wt% to 5 wt%.

11. A method according to any of the preceding claims, wherein the content comprises or consists of an oral nicotine delivery (OND) powder formulation.

12. A method of manufacturing a smokeless article for oral consumption comprising a pouch enclosing a content, the method comprising the steps of:

(i) preparing a precursor blend composition, followed by
(ii) adding a lubricant to the precursor blend composition and blending the resulting mixture to provide the content;

wherein the content satisfies one or more of the following:

(a) a Carr's Index of less than 31;
(b) a Hausner's ratio of less than 1.46;
(c) a bulk density of 0.35-0.50 g/mL;
(d) a tapped density of 0.55-0.65 g/mL;
(e) an amount of lubricant from 1 wt% to 10 wt%, for example from 1 wt% to 5 wt%, based on the total weight of the content.

13. A smokeless article for oral consumption comprising a pouch enclosing a content, the content comprising a lubricant selected from stearic acid or a salt thereof, and at least 45 wt% water, based on the total weight of the content; optionally wherein the content has a Carr's Index of less than 35, and/or a Hausner's ratio of less than 1.5.

14. A smokeless article according to claim 13, wherein the content comprises plant fibres, a nicotine source, water and flavourant.

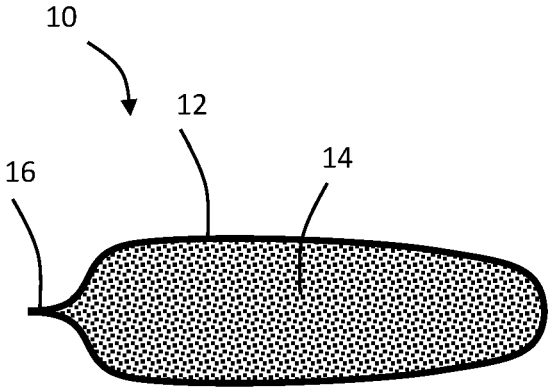

Figure 1

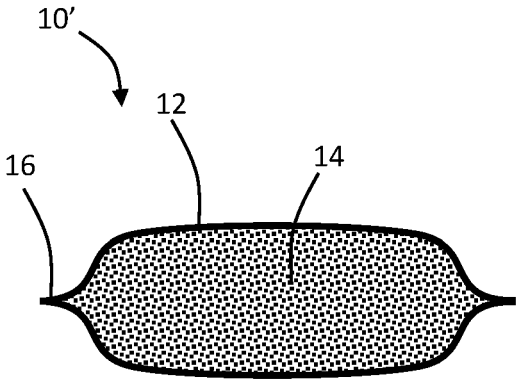

Figure 2

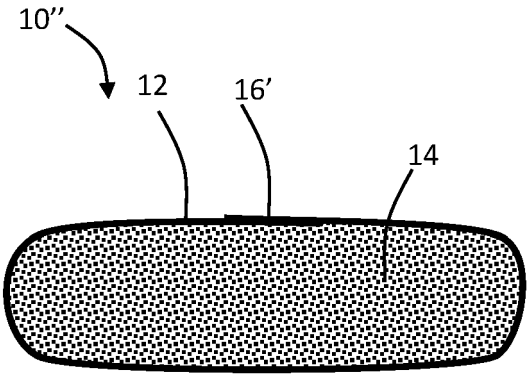

Figure 3

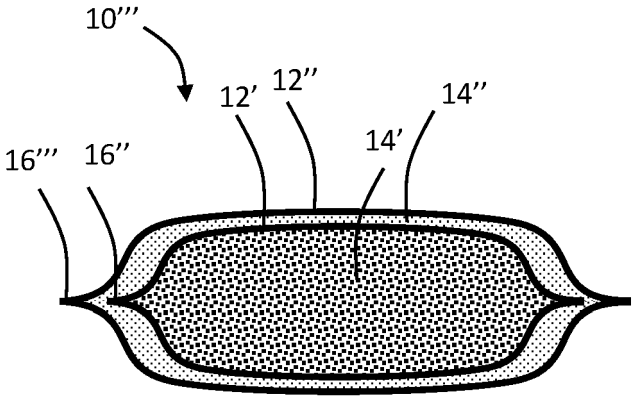

Figure 4

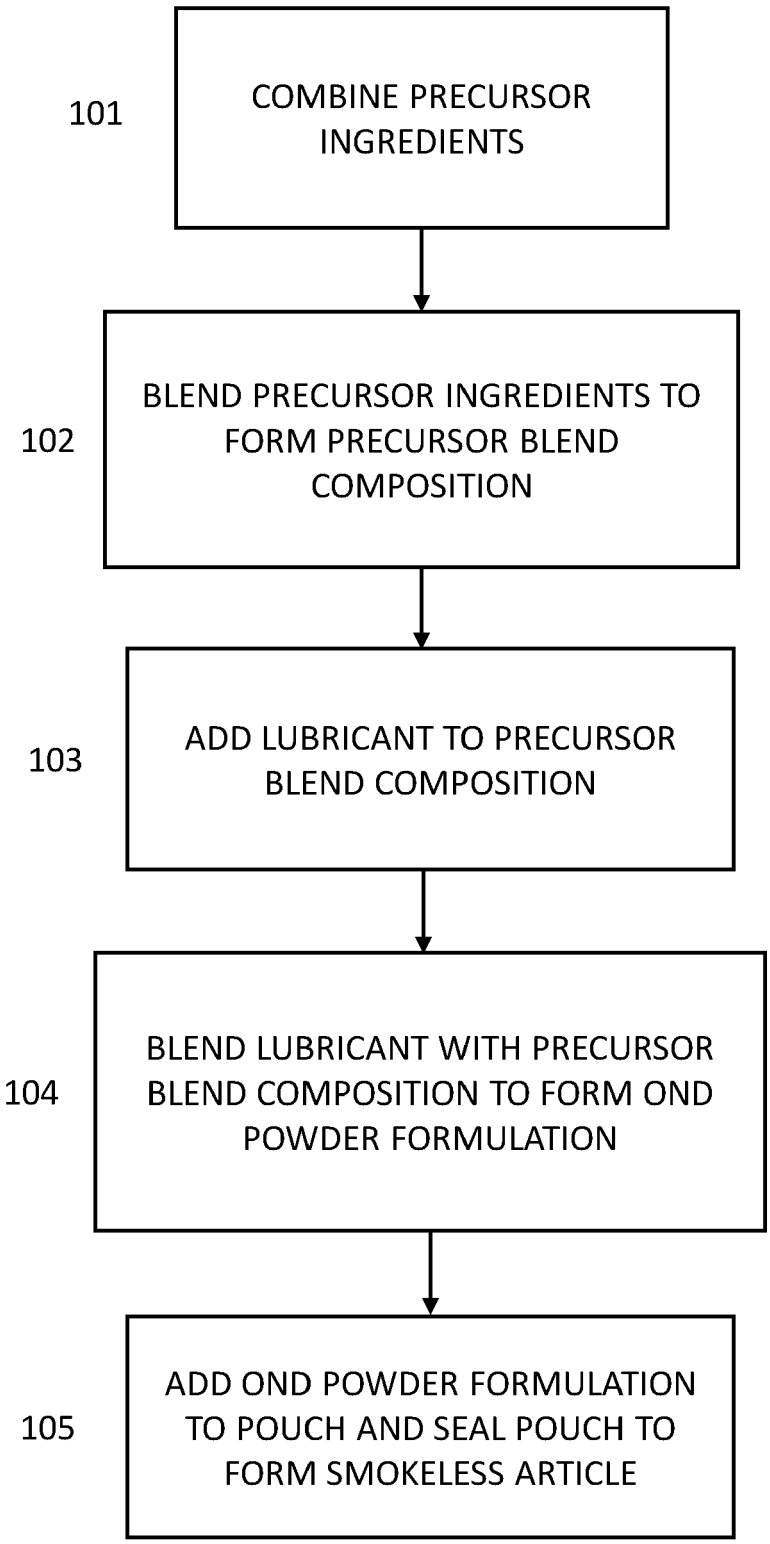
101
COMBINE PRECURSOR INGREDIENTS
102
BLEND PRECURSOR INGREDIENTS TO FORM PRECURSOR BLEND COMPOSITION
103
ADD LUBRICANT TO PRECURSOR BLEND COMPOSITION
104
BLEND LUBRICANT WITH PRECURSOR BLEND COMPOSITION TO FORM OND POWDER FORMULATION
105
ADD OND POWDER FORMULATION TO POUCH AND SEAL POUCH TO FORM SMOKELESS ARTICLE

Figure 5

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EUROPEAN SEARCH REPORT

Application Number
EP 23 19 0369

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
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A24B

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
The Hague	30 January 2024	Gzil, Piotr

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30-01-2024

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