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(54) IMPROVED SMOKING MIXTURE



(71) We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London, S.W.1, a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a smoking mixture suitable for incorporation in cigars, cigarettes and smoking pipes, in total or partial replacement of the tobacco constituent of the mixtures normally used.

Smoking mixtures normally consist mainly of, or contain a high proportion of natural tobacco and the opinion is now widely held that the smoking of tobacco, especially in cigarette form, increases the incidence of lung cancer and bronchitic ailments. The replacement of tobacco in smoking mixtures by cellulose and other smoke-producing materials has been proposed but such proposed mixtures have not been acceptable to smokers because the smoke flavour was excessively different from that of tobacco smoke.

Our co-pending Application No. 13862/70 (Serial No. 1,314,483) is directed to a smoking mixture comprising an organic combustible material as smoke-producing fuel, e.g. carbohydrate, modified carbohydrate or certain aldol condensation products, in admixture with protein. The protein serves to reduce or eliminate the intensity of the off-odours and off-flavours of the smoke from the smoke-producing material and also imparts a tobacco-like flavour and after-taste to the smoke. Such an improved smoking mixture containing protein to mask the unpleasant character of the smoke is not, however, entirely satisfactory since the protein itself can give rise to off-notes or odours.

An object of the present invention is to improve on the protein containing smoking of our co-pending Application No. 13862/70 (Serial No. 1,314,483) by including an

ingredient which masks the protein off-notes of the smoke from said mixtures.

According to the present invention there is provided a smoking mixture comprising an organic combustible material as smoke-producing fuel, protein and a conjugated polyunsaturated isoprenoid or derivative thereof such as acids and esters.

We have discovered that the conjugated polyunsaturated isoprenoids are successful in imparting a fresh vegetable-like effect to the aforementioned protein-containing smoking mixtures.

We have further discovered that if the conjugated polyunsaturated isoprenoid contains ketone rings at the end of the hydrocarbon chains then the vegetable effect is enhanced.

The smoking mixture may comprise 0.5 to 25% by weight of protein and up to 4% by weight of the polyunsaturated isoprenoid or derivative thereof, the remainder being made up of the smoke-producing fuel, additives and fillers. The preferred amount of protein is 1 to 6% by weight and the preferred amount of isoprenoid is 0.001 to 0.5% by weight.

The smoking mixtures of the invention will, in addition to protein, conjugated polyunsaturated isoprenoid and the smoke-producing fuel, contain other ingredients such as are normally used in smoking mixtures to impart desired physical properties and burning characteristics. For example, the mixtures may comprise glow-controlling catalysts, materials to improve ash coherence and colour, nicotine, flavourants, medicaments and humectants or film-forming binding agents. The mixtures may also be blended with tobacco. Such added tobacco could be in the form of comminuted tobacco or tobacco shred.

Alkali metal compounds may advantageously be used as glow-controlling catalysts and salts of ammonia, alkali metals or alkaline earth metals may be used as ash improvers.

Alkali or alkaline earth carbonates or porous fillers may be incorporated in the smoking

mixtures to give an open texture and facilitate combustion.

More particularly, additional compounds may comprise:—

- 5 1. Fillers—e.g. calcium carbonate, magnesium carbonate.
2. Humectants—e.g. glycerol, ethylene glycol, polyethylene glycol.
- 10 3. Film-forming agents—e.g. methyl cellulose, sodium carboxymethyl cellulose, pectins, gums.
4. Glow-controlling catalysts—e.g. potassium citrate, calcium carbonate, magnesium carbonate.
- 15 5. Ash cohesion agents—e.g. citric acid, sodium hydrogen phosphate, tobacco extracts.
6. Solanesol, and other similar unconjugated polyisoprenoids and derivatives thereof (e.g. esters, acids and hydroxyl derivatives), may be added to obtain a sweetish aroma from the burning mixture and to give a pleasant after-note in the mouth of the smoker.

25 The smoking mixture containing modified carbohydrate and aldol condensation product may additionally contain as a minor component one or more carbohydrate materials such as cellulose fibre, starch or sugar, to improve flavour and physical properties.

30 The conjugated polyunsaturated isoprenoid is preferably a carotenoid. It may also be a Vitamin A derivative.

The carotenoid is preferably carotene or a carotene derivative. Suitable carotenoids include α -carotene, β -carotene, admixtures of α - and β -carotene with γ -carotene, lycopene, 15,15¹ - dehydro - β - carotene, 4,4¹ - diketo-15,15¹ - dehydro - β - carotene, bixin, apocarotenal, apocarotenoid acid derivatives, canthaxanthin or other xanthophylls.

A naturally occurring isomeric mixture consisting of 85% β -carotene and 15% α -carotene may advantageously be used but pure α - or pure β -carotene is also suitable.

45 The smoke-producing fuel may be a carbohydrate material.

The carbohydrate material may advantageously comprise α -cellulose, cellulose derivatives, sugars, starch, alginate, pectin or natural gum.

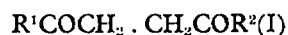
50 The smoke-producing fuel may be a modified carbohydrate.

The modified carbohydrate may advantageously be prepared as disclosed in our U.K. Patent No. 1,113,979 by subjecting it to a catalysed degradation process at a temperature of 100—250°C until the weight of the degraded material is less than 90% of the dry weight of the original carbohydrate.

60 Preferred degradation catalysts include sulphuric acid, sulphamic acid and ammonium sulphamate, and the preferred modified carbohydrate is thermally degraded cellulose.

Oxidised cellulose may also be used as an example of a modified carbohydrate. The

smoke-producing fuel may be an aldol condensation product prepared as described in our co-pending U.K. Patent Application No. 22270/69 by acid or base catalysed condensation of a compound of the formula



(or a precursor thereof), wherein R^1 and R^2 , which may be the same or different, each represents a hydrogen atom, or an alkyl, hydroxyalkyl or formyl group.

75 Preferably, the aldol condensation product comprises the condensate from succinaldehyde, acetonyl acetone or from precursors of (I) which are compounds containing a furan ring structure.

The protein should preferably be pure and it is desirable, but not essential, that any residual fat should be removed from the protein, for example, by extraction with an organic solvent. Especially valuable proteins include animal protein, for example, albumin, casein, gelatine, peptone, haemoglobin or wool protein and vegetable proteins, for example, protein from maize (zein), wheat (gluten and gliadin), soyabean or groundnut.

90 Protein acid hydrolysates and protein enzyme hydrolysates, e.g. casein acid hydrolysate and casein enzyme hydrolysate, are also suitable for the purpose of the invention.

The ratio of protein to smoke-producing fuel in the smoking mixture is in the range 1:1 to 1:60 preferably 1:8 to 1:30.

The smoking mixture is preferably prepared in a form simulating tobacco. Thus it is preferred to form the mixed ingredients into a sheet and cut or shred the sheet into the physical form required. If the carbohydrate or modified carbohydrate used is already sheeted, the smoking mixture may be prepared by merely treating the sheet with the remaining ingredients. In the usual case, where the carbohydrate is in divided form, the ingredients may be admixed and subsequently sheeted. To facilitate sheeting it is advantageous to incorporate a solution of a binding agent, for example, water-soluble cellulose ether, polyvinyl alcohol or a water-soluble gum, in the mixture.

115 The carotene or other conjugated polyunsaturated isoprenoid is advantageously sprayed from solution on to the shredded substrate. Without protein, the addition of the polyunsaturated isoprenoid has little influence on the flavour of the smoke-producing substrate. The effect is thus synergistic between the isoprenoid hydrocarbon and protein.

120 The invention is further illustrated by the following Examples in which all parts and percentages are by weight. In each of the Examples the flavour and after-taste of the product was assessed by a panel of smokers. Furthermore, in each of the Examples those

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ingredients which are not soluble are to be considered as being finely divided.

Example 1

2.8 parts of glycerol, 0.8 part citric acid and 1 part of potassium citrate dissolved in 20 parts distilled water were mixed with 2 parts sodium carboxymethyl cellulose dissolved in 80 parts hot distilled water. 0.88 part of the protein casein (extracted for 48 hours with petroleum ether at 30°C), 2 parts calcium carbonate and 10.52 parts of a material made by heating α -cellulose impregnated with 0.51 part of ammonium sulphamate for up to 4 hours at 200°C were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry is cast on glass plates and dried to give a film 0.006 in. thick which is removed from the plates, cut and shredded. The shred is sprayed with a solution of 0.04 part β -carotene in 35 parts carbon tetrachloride and the solvent allowed to evaporate. When the treated shred is made into cigarettes and smoked, it shows features of a vegetable taste and after-taste which is tobacco-like.

Example 2

2.8 parts of glycerol, 0.8 part citric acid and 1 part of potassium citrate dissolved in 20 parts distilled water were mixed with 2 parts sodium carboxymethyl cellulose dissolved in 80 parts hot distilled water. 0.88 part of the protein zein (extracted for 48 hours with petroleum ether at 30°C), 2 parts calcium carbonate and 10.52 parts of a material made by heating α -cellulose impregnated with 0.51 part of ammonium sulphamate for up to 4 hours at 200°C were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry is cast on glass plates and dried to give a film 0.006 in. thick which is removed from the plates, cut and shredded. The shred is sprayed with a solution of 0.04 part β -carotene in 35 parts carbon tetrachloride and the solvent allowed to evaporate. When the treated shred is made into cigarettes and smoked, it shows features of a vegetable taste and after-taste which is tobacco-like.

Example 3

1.34 parts of glycerol and 0.78 part of potassium citrate dissolved in 4 parts distilled water were mixed with 1.92 parts of sodium carboxymethyl cellulose dissolved in 96 parts hot distilled water. 6.73 parts of magnesium carbonate (magnesite), 3.86 parts of calcium carbonate, 0.42 parts of the protein casein (extracted for 48 hours with petroleum ether at 30°C) and 4.95 parts of a material made by heating α -cellulose with ammonium sulphamate for up to 4 hours at 200°C, were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry is cast on glass plates and dried to give a film 0.006 in. thick which is removed from the glass plates, cut and shredded. The shred is sprayed with a solution of 0.02 part of β -carotene in 35 parts carbon tetrachloride and the solvent allowed to evaporate. When the treated shred is made into cigarettes and smoked, it shows features of a vegetable taste and after-taste which is tobacco-like.

Example 4

2.8 parts of glycerol, 0.8 part citric acid and 1 part of potassium citrate dissolved in 20 parts distilled water were mixed with 2 parts sodium carboxymethyl cellulose dissolved in 80 parts hot distilled water. 0.88 part of the protein casein (extracted for 48 hours with petroleum ether at 30°C), 2 parts calcium carbonate and 10.52 parts of a material made by heating α -cellulose impregnated with 0.51 part of ammonium sulphamate for up to 4 hours at 200°C were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry is cast on glass plates and dried to give a film 0.006 in. thick which is removed from the plates, cut and shredded. The shred is sprayed with a solution of 0.04 part vitamin A acetate in 35 parts carbon tetrachloride and the solvent allowed to evaporate. When the treated shred is made into cigarettes and smoked, it shows features of a vegetable taste and after-taste which is tobacco-like.

Example 5

2.8 parts of glycerol, 0.8 part citric acid and 1 part of potassium citrate dissolved in 20 parts distilled water were mixed with 2 parts sodium carboxymethyl cellulose dissolved in 80 parts hot distilled water. 0.88 part of the protein casein (extracted for 48 hours with petroleum ether at 30°C), 2 parts calcium carbonate and 10.52 parts of a material made by heating α -cellulose impregnated with 0.51 part of ammonium sulphate for up to 4 hours at 200°C, were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry is cast on glass plates and dried to give a film 0.006 in. thick which is removed from the plates, cut and shredded. The shred is sprayed with a solution of 0.04 part canthaxanthin in 35 parts carbon tetrachloride and the solvent allowed to evaporate. When the treated shred is made into cigarettes and smoked, it shows features of a vegetable taste and after-taste which is tobacco-like.

Example 6

2.8 parts of glycerol, 0.8 part citric acid and 1 part potassium citrate dissolved in 20 parts distilled water were mixed with 2 parts sodium carboxymethyl cellulose dissolved in 80 parts hot distilled water. 0.88 part of the protein casein (extracted for 48 hours with

petroleum ether at 30°C), 2 parts calcium carbonate and 10.52 parts of a material made by heating α -cellulose impregnated with 0.51 part of ammonium sulphamate for up to 4 hours at 200°C were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry is cast on glass plates and dried to give a film 0.006 in. thick which is removed from the plates, cut and shredded. The shred is sprayed with a solution of 0.04 part lycopene in 35 parts carbon tetrachloride and the solvent allowed to evaporate. When the treated shred is made into cigarettes and smoked, it shows features of a vegetable taste and after-taste which is tobacco-like.

Example 7

1.34 parts of glycerol and 0.78 part of potassium citrate dissolved in 4 parts distilled water were mixed with 1.92 parts of sodium carboxymethyl cellulose dissolved in 96 parts hot distilled water. 6.73 parts of magnesium carbonate (magnesite), 3.86 parts of calcium carbonate, 0.42 part of the protein casein (extracted for 48 hours with petroleum ether at 30°C), and 4.95 parts of α -cellulose were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry is cast on glass plates and dried to give a film 0.006 in. thick which is removed from the plates, cut and shredded. The shred is sprayed with a solution of 0.04 part β -carotene in 35 parts carbon tetrachloride and the solvent allowed to evaporate. When the treated shred is made into cigarettes and smoked, it shows features of a vegetable taste and after-taste which is tobacco-like.

Example 8

2.8 parts of glycerol, 0.8 part citric acid and 1 part of potassium citrate dissolved in 20 parts distilled water were mixed with 2 parts sodium carboxymethyl cellulose dissolved in 80 parts hot distilled water. 1.4 parts of the protein casein (extracted for 48 hours with petroleum ether at 30°C), 2 parts calcium carbonate and 10.52 parts of a material made by heating α -cellulose impregnated with 0.51 part of ammonium sulphamate for up to 4 hours at 200°C were added to the above mixture and the whole stirred until homogeneous (about 1 hour). The slurry is cast on glass plates and dried to give a film 0.006 in. thick which is removed from the plates, cut and shredded. The shred is sprayed with a solution of 0.06 part β -carotene in 35 parts carbon tetrachloride and the solvent allowed to evaporate. When the treated shred is made into cigarettes and smoked, it shows features of a vegetable taste and after-taste which is tobacco-like.

Example 9

1.34 parts of glycerol and 0.78 part of potassium citrate dissolved in 4 parts distilled water were mixed with 1.92 parts of sodium carboxymethyl cellulose dissolved in 96 parts hot distilled water. 6.73 parts of magnesium carbonate (magnesite), 3.86 parts of calcium carbonate, 0.20 part of the protein casein (extracted for 48 hours with petroleum ether at 30°C) and 5.18 parts of a material made by heating α -cellulose with ammonium sulphamate for up to 4 hours at 200°C, were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry is cast on glass plates and dried to give a film 0.006 in. thick which is removed from the plates, cut and shredded. The shred is sprayed with a solution of 0.01 part of β -carotene in 5 parts carbon tetrachloride and the solvent allowed to evaporate. When the treated shred is made into cigarettes and smoked, it shows features of a vegetable taste and after-taste which is tobacco-like.

Example 10

2.8 parts of glycerol, 0.8 part citric acid and 1 part of potassium citrate dissolved in 20 parts distilled water were mixed with 2 parts sodium carboxymethyl cellulose dissolved in 80 parts hot distilled water. 0.88 part of the protein casein (extracted for 48 hours with petroleum ether at 30°C), 2 parts calcium carbonate and 10.52 parts of a material made by heating α -cellulose impregnated with 0.51 part of ammonium sulphamate for up to 4 hours at 200°C were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry is cast on glass plates and dried to give a film 0.006 in. thick which is removed from the plates, cut and shredded. The shred is sprayed with a solution of 0.8 part β -carotene in 35 parts carbon tetrachloride and the solvent allowed to evaporate. When the treated shred is made into cigarettes and smoked, it shows features of a vegetable taste and after-taste which is tobacco-like.

Example 11

1.34 parts of glycerol and 0.78 part of potassium citrate dissolved in 4 parts distilled water were mixed with 1.92 part of sodium carboxymethyl cellulose dissolved in 96 parts hot distilled water. 6.73 parts of magnesium carbonate (magnesite), 3.86 parts of calcium carbonate, 0.20 part of the protein casein (extracted for 48 hours with petroleum ether at 30°C) and 5.18 parts of a material made by heating α -cellulose with ammonium sulphamate for up to 4 hours at 200°C, were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry is cast on glass plates and dried to give a film 0.006 in. thick which is removed from the plates, cut and shredded. The shred

is sprayed with a solution of 0.001 part of β -carotene in 35 parts carbon tetrachloride and the solvent allowed to evaporate. When the treated shred is made into cigarettes and smoked, it shows features of a vegetable taste and after-taste which is tobacco-like.

Example 12

1.34 parts of glycerol and 0.78 part of potassium citrate dissolved in 4 parts distilled water were mixed with 1.92 part of sodium carboxymethyl cellulose dissolved in 96 parts hot distilled water. 6.73 parts of magnesium carbonate (magnesite), 3.86 parts of calcium carbonate, 0.38 part of the protein casein (extracted for 48 hours with petroleum ether at 30°C), and 5.0 parts of a material made by heating α -cellulose with ammonium sulphamate for up to 4 hours at 200°C were added to the above mixture and stirred until homogeneous (about 1 hour).

The slurry was cast on glass plates and dried to give a film 0.006 in. thick which was removed, from the plates, cut and shredded. The shred was sprayed with a solution of 0.02 part of a naturally occurring isomeric mixture of 15% α - and 85% β -carotene in 50 parts carbon tetrachloride and the solvent allowed to evaporate. When the treated shred was made into cigarettes, it showed features of a vegetable taste and after-taste which is tobacco-like.

Example 13

1.34 parts of glycerol and 0.78 part of potassium citrate dissolved in 4 parts distilled water were mixed with 1.92 parts of sodium carboxymethyl cellulose dissolved in 96 parts hot distilled water. 6.72 parts of magnesium carbonate (magnesite), 3.86 parts of calcium carbonate, 0.2 part of the protein casein (extracted for 48 hours with petroleum ether at 30°C), and 5.18 parts of a material made by heating α -cellulose with ammonium sulphamate for up to 4 hours at 200°C, were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry was cast on to glass plates and dried to give a film 0.006 in. thick which was removed from the plates, cut and shredded. This shred was sprayed with a solution of 0.01 part β -carotene in 35 parts carbon tetrachloride and the solvent allowed to evaporate. When the treated shred was made into cigarettes in a 50:50 blend with flue-cured tobacco shred and smoked in comparison with a similar blend not containing β -carotene, there was a marked preference for the cigarette containing the additive because of the presence of a fuller flue-cured flavour.

A similar result was obtained in similar exercises using either 70:30 or 85:15 shred/shred blends of artificial substrate and tobacco.

Example 14

1.8 parts of glycerol dissolved in 4 parts of distilled water were mixed with 2.4 parts sodium carboxymethyl cellulose dissolved in 96 parts distilled water. 5.98 parts of magnesium carbonate (magnesite), 3.44 parts of calcium carbonate, 0.3 parts of soya fibre protein, 1 part of bentonite and 5.08 parts of a material made by heating α -cellulose with ammonium sulphamate were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry was cast on glass plates and dried to give a film 0.006 in. thick. The film was removed and shredded. The shred was sprayed with a solution of 0.02 part β -carotene in 50 parts chloroform and the solvent allowed to evaporate. When the treated shred was made into cigarettes and smoked, it showed features of a vegetable taste and after-taste which are tobacco like.

Example 15

1.32 parts of glycerol dissolved in 4 parts of distilled water were mixed with 1.8 parts sodium carboxymethyl cellulose dissolved in 96 parts distilled water. 4.48 parts of magnesium carbonate (magnesite), 2.58 parts of calcium carbonate, 5.0 parts of protein casein, 0.74 parts of bentonite and 3.96 parts of a material made by heating α -cellulose with ammonium sulphamate were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry was cast on glass plates and dried to give a film 0.006 in. thick. The film was removed and shredded. The shred was sprayed with a solution of 0.02 part of β -carotene in 50 parts chloroform and the solvent allowed to evaporate. When the treated shred was made into cigarettes and smoked, it showed features of a vegetable taste and after taste.

Example 16

1.8 parts of glycerol dissolved in 4 parts distilled water were mixed with 2.4 parts sodium carboxymethyl cellulose dissolved in 96 parts distilled water. 5.98 parts of magnesium carbonate (magnesite), 3.44 parts of calcium carbonate, 0.3 parts of the protein gliadin, 1 part of bentonite and 5.08 part of a material made by heating α -cellulose with ammonium sulphamate were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry was cast on glass plates and dried to give a film 0.006 in. thick. The film was removed and shredded. The shred was sprayed with a solution of 0.02 part of β -carotene in 50 parts chloroform and the solvent allowed to evaporate. When the treated shred was made into cigarettes and smoked, it showed features of a vegetable taste and after taste.

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Example 17

1.8 parts of glycerol dissolved in 4 parts of distilled water were mixed with 2.4 parts sodium carboxymethyl cellulose dissolved in 96 parts distilled water. 5.98 parts of magnesium carbonate (magnesite), 3.44 parts of calcium carbonate, 0.3 parts of protein casein and 1 part of bentonite and 5.08 parts of oxidised cellulose, were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry was cast on glass plates and dried to give a film 0.006 in. thick. The film was removed and shredded. The shred was sprayed with a solution of 0.02 part of β -carotene in 50 parts chloroform and the solvent allowed to evaporate. When the treated shred was made into cigarettes and smoked it showed features of a vegetable taste and after taste which are tobacco-like.

Example 18

1.8 parts of glycerol dissolved in 4 parts of distilled water were mixed with 2.4 parts sodium carboxymethyl cellulose dissolved in 96 parts distilled water. 5.98 parts of magnesium carbonate (magnesite), 3.44 parts of calcium carbonate, 2.69 parts of protein casein, 1 part of bentonite and 2.68 parts a material made by heating α -cellulose with ammonium sulphate were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry was cast on glass plates and dried to give a film 0.006 in. thick. The film was removed and shredded. The shred was sprayed with a solution of 0.02 part of β -carotene in 50 parts ether and the solvent allowed to evaporate. When the treated shred was made into cigarettes and smoked, it showed features of a vegetable taste and after-taste which are tobacco-like.

Example 19

1.8 parts of glycerol dissolved in 4 parts of distilled water were mixed with 2.4 parts sodium carboxymethyl cellulose dissolved in 96 parts distilled water. 5.98 parts of magnesium carbonate (magnesite), 3.44 parts of calcium carbonate, 0.088 parts of protein casein and 1 part of bentonite and 5.30 parts of a material made by heating α -cellulose with ammonium sulphamate were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry was cast on glass plates and dried to give a film 0.006 in. thick. The film was removed and shredded. The shred was sprayed with a solution of 0.02 part of β -carotene in 50 parts ether and the solvent allowed to evaporate. When the treated shred was made into cigarettes and smoked it showed features of a vegetable taste and after-taste which are tobacco-like.

Example 20

1.8 parts of glycerol dissolved in 4 parts distilled water were mixed with 2.4 parts sodium carboxymethyl cellulose dissolved in 96 parts distilled water. 5.98 parts of magnesium carbonate (magnesite), 3.44 parts of calcium carbonate, 0.3 part of protein casein, 1 part of bentonite and 5.08 parts of a condensate of succinaldehyde (U.K. Patent Application No. 22270/69 (Serial No. 1,298,354) were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry was cast on glass plates and dried to give a film 0.006 in. thick. The film was removed and shredded. The shred was sprayed with a solution of 0.02 part of β -carotene in 50 parts chloroform and the solvent allowed to evaporate. When the treated shred was made into cigarettes and smoked, it showed features of a vegetable taste and after-taste which are tobacco-like.

Example 21

1.8 parts of glycerol dissolved in 4 parts of distilled water were mixed with 2.4 parts sodium carboxymethyl cellulose dissolved in 96 parts distilled water, 5.98 parts of magnesium carbonate (magnesite), 3.44 parts of calcium carbonate, 0.3 parts of protein casein, 1 part of bentonite and 5.08 parts of a material made by heating α -cellulose with ammonium sulphamate were added to the above mixture and the whole stirred until homogenous (about 1 hour).

The slurry was cast on glass plates and dried to give a film 0.006 in. thick. The film was removed and shredded. The shred was sprayed with a solution of 0.04 parts of apocarotenal in 50 parts chloroform and the solvent allowed to evaporate. When the treated shred was made into cigarettes and smoked in comparison with a similar cigarette not containing the sprayed on additive, there was a marked preference for the cigarette with the additive because of improved tobacco character.

Example 22

1.8 parts of glycerol dissolved in 4 parts of distilled water were mixed with 2.4 parts sodium carboxymethyl cellulose dissolved in 96 parts distilled water, 5.98 parts of magnesium carbonate (magnesite), 3.44 parts of calcium carbonate, 0.3 parts of protein casein, 1 part of bentonite and 5.08 parts of a material made by heating α -cellulose with ammonium sulphamate were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry was cast on glass plates and dried to give a film 0.006 in. thick. The film was removed and shredded. The shred was sprayed with a solution of 0.4 part Vitamin A acetate in 50 parts chloroform and the

solvent allowed to evaporate. When the treated shrew was made into cigarettes and smoked in comparison with a similar cigarette not containing the sprayed on additive, there was a marked preference for the cigarettes with the additive because of improved tobacco character.

Example 23

0.34 parts glycerol were dissolved in 4 parts distilled water and mixed with 6.6 parts sodium carboxymethyl cellulose dissolved in 96 parts distilled water. 6.2 parts of dolomite (Dolodust) 5.6 parts of diatomaceous earth (Celite) 0.22 parts charcoal, 0.34 parts octanol and 0.4 part casein protein were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry was cast on to glass plates and dried to give a film 0.006 in. thick. The film was removed and shredded and the shred was sprayed with a solution of 0.02 parts β -carotene in 50 parts chloroform and the solvent allowed to evaporate. When the treated shred was made into cigarettes and smoked in comparison with a similar cigarette not containing the sprayed on additive, there was a marked preference for the cigarette with the additive because of improved taste and after-taste which had a cigar/pipe tobacco nature.

WHAT WE CLAIM IS:—

1. A smoking mixture comprising an organic combustible material as smoke-producing fuel, protein and a conjugated polyunsaturated isoprenoid or derivatives thereof such as acids or esters.

2. A smoking mixture as claimed in Claim 1 comprising 0.5 to 25% by weight protein and up to 4% by weight of the polyunsaturated isoprenoid or derivative thereof.

3. A smoking mixture as claimed in Claim 1 or Claim 2 comprising 1 to 6% by weight protein and 0.001 to 0.5% by weight isoprenoid.

4. A smoking mixture as claimed in any one of the preceding claims wherein the ratio of protein to smoke-producing fuel is 1:1 to 1:60.

5. A smoking mixture as claimed in Claim 4 wherein the ratio of protein to smoke-producing fuel is 1:8 to 1:30.

6. A smoking mixture as claimed in any one of the preceding claims wherein the smoke-producing fuel is carbohydrate.

7. A smoking mixture as claimed in Claim 6 wherein the carbohydrate is α -cellulose, cellulose derivatives, sugars, starch, alginate, pectin or natural gum.

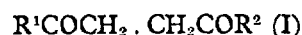
8. A smoking mixture as claimed in any one of Claims 1 to 5 wherein the smoke-producing fuel is a modified carbohydrate.

9. A smoking mixture as claimed in Claim 8 wherein the modified carbohydrate is oxidised cellulose.

10. A smoking mixture as claimed in Claim 8 wherein the modified carbohydrate is thermally degraded carbohydrate prepared as disclosed in U.K. Patent No. 1,113,979 by subjecting carbohydrate to a catalysed degradation process at a temperature of 100—250°C until the weight of the degraded material is less than 90% of the dry weight of the original carbohydrate.

11. A smoking mixture as claimed in Claim 10 wherein the modified carbohydrate is thermally degraded cellulose.

12. A smoking mixture as claimed in any one of Claims 1 to 5 wherein the smoke-producing fuel is a solid condensate produced by acid or base catalysed condensation of a compound of the formula



(or a precursor thereof), wherein R^1 and R^2 which may be the same or different, each represents a hydrogen atom, or an alkyl, hydroxyalkyl or formyl group.

13. A smoking mixture as claimed in Claim 12 wherein the smoke-producing fuel comprises the condensate from succinaldehyde, acetonyl acetone or from precursors of (I) which are compounds containing a furan ring structure.

14. A smoking mixture as claimed in any one of the preceding claims wherein the conjugated polyunsaturated isoprenoid is a carotenoid or a Vitamin A derivative.

15. A smoking mixture as claimed in Claim 14 wherein the carotenoid is carotene or a carotene derivative.

16. A smoking mixture as claimed in Claim 14 wherein the carotenoid is α -carotene, β -carotene, admixture of α - and β -carotene with γ -carotene, lycopene, 15,15¹ - dehydro - β -carotene, 4,4¹ - diketo - 15,15¹ - dehydro - β -carotene, bixin, apocarotenal, apocarotenoic acid derivatives, canthaxanthin or other xanthophylls.

17. A smoking mixture as claimed in Claim 14 wherein the carotenoid is a naturally occurring isomeric mixture of α - and β -carotene.

18. A smoking mixture as claimed in any one of the preceding claims wherein the protein is pure and free of residual fat.

19. A smoking mixture as claimed in any one of the preceding claims wherein the protein is an animal protein such as albumin, casein, gelatine, peptone, haemoglobin or wool protein or a vegetable protein such as protein from maize (zein), wheat (glutin and gliadin), soyabean or groundnut.

20. A smoking mixture as claimed in any one of the preceding claims comprising protein acid hydrolysates or protein enzyme hydrolysates.

21. A smoking mixture as claimed in Claim

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solvent allowed to evaporate. When the treated shrew was made into cigarettes and smoked in comparison with a similar cigarette not containing the sprayed on additive, there was a marked preference for the cigarettes with the additive because of improved tobacco character.

Example 23

0.34 parts glycerol were dissolved in 4 parts distilled water and mixed with 6.6 parts sodium carboxymethyl cellulose dissolved in 96 parts distilled water. 6.2 parts of dolomite (Dolodust) 5.6 parts of diatomaceous earth (Celite) 0.22 parts charcoal, 0.34 parts octanol and 0.4 part casein protein were added to the above mixture and the whole stirred until homogeneous (about 1 hour).

The slurry was cast on to glass plates and dried to give a film 0.006 in. thick. The film was removed and shredded and the shred was sprayed with a solution of 0.02 parts β -carotene in 50 parts chloroform and the solvent allowed to evaporate. When the treated shred was made into cigarettes and smoked in comparison with a similar cigarette not containing the sprayed on additive, there was a marked preference for the cigarette with the additive because of improved taste and after-taste which had a cigar/pipe tobacco nature.

WHAT WE CLAIM IS:—

1. A smoking mixture comprising an organic combustible material as smoke-producing fuel, protein and a conjugated polyunsaturated isoprenoid or derivatives thereof such as acids or esters.

2. A smoking mixture as claimed in Claim 1 comprising 0.5 to 25% by weight protein and up to 4% by weight of the polyunsaturated isoprenoid or derivative thereof.

3. A smoking mixture as claimed in Claim 1 or Claim 2 comprising 1 to 6% by weight protein and 0.001 to 0.5% by weight isoprenoid.

4. A smoking mixture as claimed in any one of the preceding claims wherein the ratio of protein to smoke-producing fuel is 1:1 to 1:60.

5. A smoking mixture as claimed in Claim 4 wherein the ratio of protein to smoke-producing fuel is 1:8 to 1:30.

6. A smoking mixture as claimed in any one of the preceding claims wherein the smoke-producing fuel is carbohydrate.

7. A smoking mixture as claimed in Claim 6 wherein the carbohydrate is α -cellulose, cellulose derivatives, sugars, starch, alginate, pectin or natural gum.

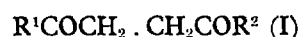
8. A smoking mixture as claimed in any one of Claims 1 to 5 wherein the smoke-producing fuel is a modified carbohydrate.

9. A smoking mixture as claimed in Claim 8 wherein the modified carbohydrate is oxidised cellulose.

10. A smoking mixture as claimed in Claim 8 wherein the modified carbohydrate is thermally degraded carbohydrate prepared as disclosed in U.K. Patent No. 1,113,979 by subjecting carbohydrate to a catalysed degradation process at a temperature of 100—250°C until the weight of the degraded material is less than 90% of the dry weight of the original carbohydrate.

11. A smoking mixture as claimed in Claim 10 wherein the modified carbohydrate is thermally degraded cellulose.

12. A smoking mixture as claimed in any one of Claims 1 to 5 wherein the smoke-producing fuel is a solid condensate produced by acid or base catalysed condensation of a compound of the formula



(or a precursor thereof), wherein R^1 and R^2 which may be the same or different, each represents a hydrogen atom, or an alkyl, hydroxyalkyl or formyl group.

13. A smoking mixture as claimed in Claim 12 wherein the smoke-producing fuel comprises the condensate from succinaldehyde, acetyl acetone or from precursors of (I) which are compounds containing a furan ring structure.

14. A smoking mixture as claimed in any one of the preceding claims wherein the conjugated polyunsaturated isoprenoid is a carotenoid or a Vitamin A derivative.

15. A smoking mixture as claimed in Claim 14 wherein the carotenoid is carotene or a carotene derivative.

16. A smoking mixture as claimed in Claim 14 wherein the carotenoid is α -carotene, β -carotene, admixture of α - and β -carotene with γ -carotene, lycopene, 15,15¹-dehydro- β -carotene, 4,4¹-diketo-15,15¹-dehydro- β -carotene, bixin, apocarotenal, apocarotenoid acid derivatives, canthaxanthin or other xanthophylls.

17. A smoking mixture as claimed in Claim 14 wherein the carotenoid is a naturally occurring isomeric mixture of α - and β -carotene.

18. A smoking mixture as claimed in any one of the preceding claims wherein the protein is pure and free of residual fat.

19. A smoking mixture as claimed in any one of the preceding claims wherein the protein is an animal protein such as albumin, casein, gelatine, peptone, haemoglobin or wool protein or a vegetable protein such as protein from maize (zein), wheat (glutin and gliadin), soyabean or groundnut.

20. A smoking mixture as claimed in any one of the preceding claims comprising protein acid hydrolysates or protein enzyme hydrolysates.

21. A smoking mixture as claimed in Claim

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20 comprising casein acid hydrolysate or casein protein hydrolysate.

5 22. A smoking mixture as claimed in any of the preceding claims comprising glow-controlling catalysts, materials to improve ash coherence and colour, nicotine, flavourants, medicaments and humectants or film forming building agents.

23. A smoking mixture as claimed in any

one of the preceding claims blended with 10 tobacco.

24. A smoking mixture substantially as hereinbefore described with reference to the Examples.

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