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DENTIFRICE COMPOSITION

The present invention relates to a dentifrice composition. In particular, it relates to a dentifrice composition having desirable rheological properties suitable for efficient filling into a mechanically operated or pressure differential dentifrice dispenser and extrusion therefrom.

A dentifrice is generally recognisable by its creamy or gel consistency and may commonly be called a dental cream, a toothpaste or, in some cases, a clear gel or opacified gel toothpaste. Indeed, it can be characterised as semi-solid, for instance, being essentially solid when standing on the bristles of a toothbrush and essentially liquid such as during manufacture with agitation and filling into a container or when subject to pressure to extrude the dentifrice from its container.

The creamy consistency of dentifrices is typically imparted by a gelling or binding agent. In the past, gelling agents have been selected primarily to provide ease of dispersion of the dentifrice in the oral cavity. Many gelling agents such as cellulosic materials, seaweed derivatives, gums and clays meet this criteria. However, some gelling agents while generally desirable for dentifrices packaged in flexible tubes, do cause disadvantages when the dentifrices are packaged in mechanically operated or pressure differential dispensers.

Dentifrices containing conventional polishing

- agents such as alpha-alumina trihydrate and conventional gelling agents such as sodium carboxymethyl cellulose or hydroxyethyl cellulose or mixtures thereof can be desirably filled into flexible tubes or mechanically operated or pressure differential dispensers and extruded therefrom. However, with many of the active ingredients often used in dentifrices difficulties occur when attempting to fill a dentifrice into a mechanically operated or pressure differential
5. dispenser and/or when attempting to extrude it therefrom. In particular, leakage from orifices in the dispensers may occur during filling or shipping and the product can be difficult to extrude in rheologically desirable ribbon form.
10. Dentifrices which are thick or tend to thicken may become increasingly difficult for a consumer to extrude from a dentifrice tube over a period of time. In other words, the consumer may have to increase pressure on a dentifrice tube containing such a
15. dentifrice during the period of use in order to soften or liquify the semi-solid dentifrice mass to extrude it. This has not been a major problem in the past since formulations can be adjusted to use less gelling agent to strike a balance of not being too soft at the
20. start of use and not too thick at the end and in any event consumers have readily adjusted to applying the amount of pressure necessary to extrude the desired amount of dentifrice on to bristles of a toothbrush.
25. Dentifrices which are thin upon extrusion from a tube have also been tolerated during use if they set to
- 30.

more solid form on toothbrush bristles within a few seconds. Thus, the type of gelling agent could be widely varied for dentifrices packaged in tubes.

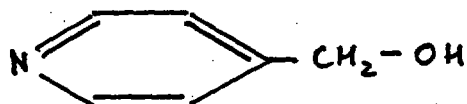
Indeed, the Copenhagen Pectin Factory Ltd., of Little

5. Skensved, Denmark, a subsidiary of Hercules Inc., of Wilmington, Delaware, U.S.A., has proposed its product Genuvisco Type 0819, an iota-carrageenan (i-carrageenan), as a possible thickener for toothpaste. I-carrageenan available from Marine Colloids
10. Division of FMC Corp., of Springfield, New Jersey, U.S.A., as Viscarin TP-5 has also been proposed for possible toothpaste use with toothpaste containing dicalcium phosphate or silica. Moreover, i-carrageenan has been disclosed as a thickener component together
15. with k-carrageenan and alkali metal alginate for a dentifrice containing galactan galactose in Japanese Patent Publication 56 115711, published 11 September 1981, of Lion Dentifrice Ltd.

- When a dentifrice dispenser which operates
20. mechanically or by pressure differential is employed, conventional techniques of reducing thickening of dentifrice may not be fully satisfactory since compared with a dentifrice filled into a flexible tube, when a dispenser is used the dentifrice must be relatively
 25. thin during filling but regain its consistency and remain so during the periods of shipping, storage and use. In other words, the dentifrice after filling essentially regains the consistency it had prior to the liquifying effect during shear filling for shipping,
 30. storage and use. Conventional gelling agent such as

- sodium carboxymethyl cellulose, hydroxyethyl cellulose or mixtures thereof may be desirably used for dentifrice containing alpha-alumina trihydrate polishing agent which is to be filled into a dispenser. However,
5. when such a dentifrice contains a number of active ingredients, such as at least two sources of fluorine, a desensitising agent such as allantoin ($C_7H_6N_4O_3$) and a vasodilator agent such as pyridyl carbinol,

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15. difficulties occur when the dentifrice is filled into a mechanically operated or pressure differential dispenser. In particular, dentifrice may seep through the space between a rod and a piston which are used particularly in a mechanically operated dispenser and
20. leakage will then occur at dispenser orifices. Further, the dentifrice ribbon extruded from a mechanically operated or pressure differential dispenser may be irregular or non-continuous. Since it is particularly desirable to prepare a dentifrice containing alpha-
25. alumina trihydrate polishing agent which includes several active agents, such as those indicated above, due to compatibility of such materials with the polishing agent, an alternative gelling system was needed.
30. Unexpectedly from amongst alternative types of gelling agents a mixture of a cellulosic gelling agent

and i-carrageenan provides an excellent dentifrice containing alpha-alumina trihydrate and several active ingredients which is to be filled into and extruded from a mechanical or pressure differential dispenser.

5 It is noteworthy that xanthan has not been compatible with cellulosic gelling agents since it may contain cellulase.

It is an advantage of the present invention that a dentifrice is provided which is readily filled into and extrudable from a mechanically operated or pressure differential dentifrice dispenser.

In accordance with the present invention a dentifrice adapted to be packaged in a mechanically operated or pressure differential dispenser comprises

15 20-80% by weight of an aqueous humectant vehicle, 0.1-5% by weight of gelling agent mixture, 20-75% by weight of alpha-alumina trihydrate polishing agent, sodium fluoride and sodium monofluorophosphate in amount to provide 300 to 10000 ppm by weight of

20 fluorine, 0.05-0.5% by weight of allantoin desensitising agent and 0.05-0.5% by weight of pyridyl carbinol vasodilator agent, wherein said gelling agent mixture is a mixture of a cellulosic gelling agent or xanthan and i-carrageenan in a weight ratio either of

25 cellulosic gelling or xanthan to i-carrageenan of 5:1 to 1:5, preferably 3:1 to 1:3, subject to the proviso that the total of all the ingredients is 100% by weight.

In the dentifrice formulation the dental vehicle

30 comprises a liquid phase proportioned with the gelling

agent to form an extrudable creamy mass of desirable consistency. The liquid phase in the dentifrice will comprise chiefly water and humectant such as polyols including glycerine, sorbitol, maltitol, xylitol, low molecular weight polyethylene glycol (e.g. 400 or 600), and propylene glycol including suitable mixtures thereof. It is advantageous usually to use as the liquid phase water and a humectant such as glycerine, sorbitol or polyethylene glycol, typically in amounts of 10-55% by weight of water and 20-55% by weight of humectant.

The dentifrice contains alpha-alumina trihydrate polishing agent in amounts of 20-75% by weight. Alpha-alumina trihydrate is typically prepared by the Bayer process and used as particles below 40 microns, principally 3-20 microns, in size. Other polishing agents may be included such as insoluble sodium metaphosphate, dicalcium phosphate, calcined alumina, silica or calcium carbonate in minor amount with respect to alpha-alumina trihydrate.

The gelling agent mixture of the present invention is present in the dentifrice in an amount of 0.1-5% by weight. It contains either a cellulosic gelling agent or xanthan mixed with i-carrageenan wherein the weight ratio of the said cellulosic gelling agent or xanthan to the said i-carrageenan is from 5:1 to 1:5, preferably from 3:1 to 1:3, and the total gelling agent mixture is preferably 0.2-3% by weight of the dentifrice.

As mentioned above, iota-carrageenan is commercially available as Genuvisco type 0819 and Viscarin TP-5 and has been recommended for use in a toothpaste. Such use in a toothpaste was described in Japanese

5. Patent Publication 56 115711 of Lion Dentifrice Ltd., wherein i-carrageenan is mentioned as a possible component of a gelling system together with k-carrageenan and alkali metal alginate. In U.S. Patent 4,353,890 to Scott, i-carrageenan is disclosed as an alternative
10. to k-carrageenan as a toothpaste gelling agent wherein the toothpaste is subjected to microwave radiation to reduce the tendency of carrageenan in general to become thin during manufacture. The carrageenan may be the sole gelling agent or mixed with other gelling agents.
15. In the present invention, dentifrice containing i-carrageenan to be filled into a mechanically operated or pressure differential dispenser does not require microwave radiation.

- The prior art generally discussed above does not
20. indicate that gelling systems of cellulose gelling agent and i-carrageenan can provide dentifrices containing alpha-alumina trihydrate polishing agent and many active ingredients with the properties necessary for efficient filling into mechanically operated or
 25. pressure differential dispenser and extrusion therefrom.

- It is noteworthy that U.S. Patent 4,029,760 to de Roeck et al., discloses an oral composition in which i-carrageenin is set forth as an antigingivitis agent alternative to other carrageenins. Carrageenins are
30. highly depolymerised derivatives of carrageenans.

Carrageenans do not appear to provide an antigingivitis effect.

Dentifrices are commonly manufactured by cold process, e.g. at about 25°C, or by a hot process, e.g. at about 60°C. I-carrageenan can be used in either cold process or hot process techniques. Since xanthan is made with cold processing, when it is employed, it may be readily mixed with i-carrageenan and incorporated together therewith in the dentifrice by cold processing.

Physical properties of Genuvisco type 0819 i-carrageenan are indicated below:

1. Viscosity of 0.30% by weight solution of GENUVISC0 type 0819 in lean solvent (high glycerine, low water) prepared using a hot process (60°C):
 Viscosity = 110 ± 17 cP at 32 rpm.
 Viscosity = 70 ± 11 cP at 61 rpm.
 Viscosity = 45 ± 7 cP at 128 rpm.
 Measured on HAAKE Rotovisco RV3 at 25°C.
2. Viscosity of 0.30% by weight solution of GENUVISC0 type 0819 in lean solvent prepared using a cold process (25°C).
 Viscosity = 450 ± 60 cP measured on Brookfield Viscometer LVT at 25°C.
 Viscosity = 85 ± 13 cP at 32 rpm.
 Viscosity = 55 ± 8 cP at 64 rpm.
 Viscosity = 37 ± 6 cP at 128 rpm.
 Measured on HAAKE Rotovisco RV3 at 25°C.

3. Particle size: Less than 1% by weight gum on 0.075 mm test sieve (DIN 80,200 U.S. mesh).
- 5 4. Moisture content: Less than 12% by weight.
5. pH: 8.5 $\pm$ 1.5 in 0.5% by weight solution in distilled water at 25°C.
6. Colour: White to cream.

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Viscarin TP-5 i-carrageenan has the following physical properties:

- Colour: light tan to tan;
- 15 Particle size: more than 95.0% by weight through a U.S. Standard Sieve, 250 nm (series #60).
- Moisture: maximum 12.0% by weight (Cenco Moisture Balance).
- 20 pH: 7.0 to 9.5, 1.5% by weight solution, 30°C.

Typical desirable cellulosic gelling agents include alkali metal carboxymethyl cellulose, methyl cellulose, 25 hydroxymethyl cellulose and hydroxypropyl cellulose. Sodium carboxymethyl cellulose is preferred. The mixed gel system is present in amount of 0.1-5% by weight of the dentifrice, and the weight ratio of cellulosic gelling agent to i-carrageenan is from 5:1 to 1:5, 30 preferably 3:1 to 1:3 and most preferably about 1:1. Thus, a typical preferred dentifrice can contain 0.9-1.2% total gelling agent including 0.3-0.9% of each

of cellulosic gelling agent and i-carrageenan, the weight ratio between the two being from 3:1 to 1:3.

It is noteworthy that grades of sodium carboxymethyl cellulose which may be used include the following:

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TABLE 1

	<u>SUPPLIER</u>	<u>CMC GRADE</u>	<u>VISCOSITY</u>
	Hercules	7MXF	300-500
	Hercules	7MFD	300-500
10	Hercules	9M31F	900-1200
	Hercules	9M31XF	900-1200
	Hercules	12M31XF	900-1200
	Hercules	7MF	300-500
	Hercules	12M31PD	900-1200
15	Hercules	7M8SXF	200-800
	Wolff Walsrode	Walocel CRT 1000 PA 07	700-1200
	Nyma	Nymcel (Registered Trade Mark) ZMF.33*	50-80
	Enka	Akucell AC 1642*	80-120
20	Enka	Akucell AC 1632*	60-120
	Cros	Cellogen (Registered Trade Mark) HP-SA	700-900
	Uddeholm	Cekol MVEP	500-800
	Hoechst	Tylose (Registered Trade Mark) CB 200**	120-260
25			

measured in 1.5% by weight solution except:

* 1% solution (Brookfield; 25°C).

30 ** Hoeppler Viscometer (2% by weight; 20°C).

Further, grades of hydroxyethyl cellulose which may be used include the following:

TABLE 2

<u>SUPPLIER</u>	<u>HEC GRADE</u>	<u>VISCOSITY</u>
Hercules	Natrosol (Registered	
	Trade Mark) 250M	
5.	and MR	4500-6500
Hercules	Natrosol 250 HR*	
	and 250 H*	1500-2500
Hercules	Natrosol 250 HHR*	
	and 250 HH	3400-5000
10.	BP Chemicals	Cellobond (Registered
	Trade Mark) 5000 A	4200-5600
	BP Chemicals	Cellobond 7000 A
		6000-7000
	Hoechst	Tylose H 4000 P**
		3000-5000
	Hoechst	Tylose H 10000 P**
		700-1200

15.

* 1% by weight solution (Brookfield; 25°C).

** Hoesppler Viscometer (2% by weight; 25°C).

- The i-carrageenan and cellulosic gelling agent may be
20. mechanically mixed together prior to mixing with the liquid phase of the dental cream vehicle or may be mixed separately with the liquid phase, with hot processing (typically about 60°C) or cold processing (typically about 25°C) techniques.
25. Xanthan gum is a fermentation product prepared by action of the bacteria of the genus *Xanthomonas* upon carbohydrates. Four species of *Xanthomonas*, viz *X campetris*, *X phaseolei*, *X malvoearum* and *X carotae* are reported in the literature to be the most efficient
30. gum producers. Although the exact chemical structure is not determined, it is generally accepted to be a heteropolysaccharide with a molecular weight of several million. It contains D-glucose, D-mannose

and D-glucuronic acid in the molar ratio of 2.8:3:2.0. The molecule contains 4.7% by weight acetyl and about 3% by weight pyruvate. The proposed chemical structure configuration can be found in McNeely and Kang,

5. Industrial Gums, ed. R.L. Whistler, CH XXI, 2nd Edition, New York, 1973. The procedure for growing, isolating and purifying the xanthan gum is also found in that publication. Further description of xanthan gum is found in Manufacturing Chemist, May 1960, pages
10. 206-208 (including mention at page 208 of potential use of gums therein described for formulating toothpastes).

The i-carrageenan and xanthan may be mechanically mixed together prior to mixing with the liquid phase of the dental cream vehicle or may be mixed separately

15. with the liquid phase, with cold processing (typically about 25°C) techniques.

The dentifrice is packaged in a container from which it can be readily extruded such as a pressure differential or mechanically operated dental cream

20. dispenser. These dental cream dispensers are differentiated from ordinary toothpaste tubes in that the dispenser has an outer rigid wall and the force required to dispense the product is generated within the container. This may be by mechanical pump action
25. in a mechanically operated dental cream dispenser or by gas pressure or atmospheric pressure in a pressure differential dispensing container of the aerosol or vacuum type respectively. The rheological properties are highly desirable when a mechanically operated
30. dispensing container of the type described in British Patent Application 2 070 695A, published 9 September 1981, is employed. This dispensing container comprises a dispensing mouthpiece, a tension member, a central

rod, a piston and operating hand control.

Examples of pressure differential dispensing containers include the aerosol and vacuum type.

Suitable pressure differential dispensers include those

5. comprising a collapsible product-containing bag being disposed within a rigid container which contains a propellant fluid. In such dispensing containers, operation of the valve permits release of the product only, the propellant fluid being separated from the
10. product by the fluid impermeable bag. Dispensers of this type are described in U.S. Patents 3,828,977 and 3,838,976. These are so-called Sepro dispensers. So-called Exxel containers also utilise pressure.

Still another type dispenser is the barrier

15. piston container described in U.S. 4,171,757. Such container includes a valve, a product-containing compartment and an essentially fluid-tight barrier piston which separates the propellant fluid from the contained product (the so-called Diamond container).

20. A process for preparing a dispenser containing a dentifrice in accordance with the present invention preferably comprises extruding into a mechanically operated dispenser an amount of the said dentifrice through a nozzle to fill the dispenser which is open at
25. its bottom and which contains a central rod, sliding into place around the said central rod a piston having a diameter corresponding to the inner diameter of the dispenser and a central hole to receive the said central rod and then sealing the said dispenser with a
30. bottom disc.

The dentifrice contains as active agents a mixture of sodium fluoride and sodium monofluorophosphate to provide 300-10000 ppm by weight of fluorine, e.g. 750-2000 ppm, and particularly 1400-2000 ppm such as 1400-1670 ppm. A binary fluoride system of sodium monofluorophosphate and sodium fluoride is desirably used in which 30-40% of the fluorine (e.g. 30-35%) is provided by sodium fluoride.

Sodium monofluorophosphate, $\text{Na}_2\text{PO}_3\text{F}$, as commercially available, may vary considerably in purity. It may be used in any suitable purity provided that any impurities do not substantially adversely affect the desired properties. In general, the purity is desirably at least 80%. For best results, it should be at least 85%, and preferably at least 90% by weight of sodium monofluorophosphate with the balance being primarily impurities or by-products of manufacture such as sodium fluoride and water-soluble sodium phosphate salt. Expressed in another way, the sodium monofluorophosphate employed should have a total fluoride content of above 12% by weight, preferably above 12.7%, a content of not more than 1.5%, preferably not more than 1.2% of free sodium fluoride; and a sodium monofluorophosphate content of at least 12%, preferably at least 12.1% all calculated as fluoride.

As indicated above, sodium fluoride in the binary mixture is a separate fluorine-containing component from sodium monofluorophosphate. 225-800 ppm by weight of fluorine is preferably provided to the dental cream by sodium fluoride.

- Additional active agent is provided to the dentifrice by the presence of 0.05-0.5% by weight, preferably 0.08-0.2%, of allantoin desensitising agent. Such agent combats sensitivity
5. of wounded periodontal tissue and promotes its healing. Allantoin can be obtained from ABM Industrial Products Ltd., of Woodley, Stockport, Cheshire, England.
- Pyridyl carbinol active agent in an amount of 0.05-0.5% by weight, preferably 0.08-0.2%, provides the
10. dentifrice with a vasodilator effect.
- When a dentifrice containing alpha-alumina trihydrate polishing agent, the binary fluoride system, allantoin desensitising agent and pyridyl carbinol vasodilator agent, described above, is employed filling
- 15.

and extrusion using mechanically operated or pressure differential dispensers is made efficient and rheologically desirable with the gel system of the present invention.

5. Filling may be effected by conventional techniques. For instance, when a mechanically operated dispenser of the type described in published British Patent Application 2 070 695A is used, a predetermined amount of the dentifrice is extruded through a nozzle
10. to fill the dispenser which is open at its bottom and which contains a central rod. A piston having a diameter corresponding to the inner diameter of the dispenser and a central hole to permit insertion of the central rod therein is slid into place. The dispenser
15. is then sealed with a bottom disc.

- Any suitable surface active or deterative material may be included in the dentifrice compositions of the present invention. Such compatible materials are desirable to provide additional deterative, foaming and
20. anti-bacterial properties depending upon the specific type of surface active material and are selected similarly. These detergents are water-soluble compounds usually, and may be anionic, nonionic or cationic in structure. It is usually preferred to use
25. the water-soluble non-soap or synthetic organic detergents. Suitable deterative materials are known and include, for example, the water-soluble salts of higher fatty acid monoglyceride monosulphate detergent (e.g. sodium coconut fatty acid monoglyceride monosulphate),
30. higher alkyl sulphate (e.g. sodium lauryl sulphate),

alkyl aryl sulphonate (e.g. sodium dodecyl benzene sulphonate, and higher fatty acid esters of 1,2 dihydroxy propane sulphonate).

Further surface active agents include the
5 substantially saturated higher aliphatic acyl amides of lower aliphatic amino carboxylic acid compounds, such as those having 12 to 16 carbon atoms in the acyl radical. The amino acid portion is derived generally from the lower aliphatic saturated monoaminocarboxylic
10 acids having 2 to 6 carbon atoms, usually the monocarboxylic acid compounds. Suitable compounds are the fatty acid amides of glycine, sarcosine, alanine, 3-amino-propanoic acid and valine having 12 to 16 carbon atoms in the acyl group. It is preferred to use
15 the N-lauroyl, myristoyl and palmitoyl sarcoside compounds, however, for optimum effects.

The amide compounds may be employed in the form of the free acid or preferably as the water-soluble salts thereof, such as the alkali metal, ammonium,
20 amine and alkylolamine salts. Specific examples thereof are the sodium and potassium N-lauroyl, myristoyl and palmitoyl sarcosides, ammonium and ethanolamine N-lauroyl glycide and alanine. For convenience herein, reference to "amino carboxylic
25 acid compound", "sarcoside", and the like refers to such compounds having a free carboxylic group or the water-soluble carboxylate salts.

Such materials are utilised in pure or substantially pure form. They should be as free as
30 practicable from soap or similar higher fatty acid

material which tends to reduce the activity of these compounds. In usual practice, the amount of such higher fatty acid materials is less than 15% by weight of the amide and insufficient to substantially
 5 adversely affect it, and preferably less than 10% of the said amide material.

Various other materials may be incorporated in the dentifrice of the present invention. Examples thereof are colouring or whitening agents,
 10 preservatives, such as methyl p-hydroxybenzoate, stabilisers, tetrasodium pyrophosphate, silicones, chlorophyll compounds and ammoniated materials such as urea, diammonium phosphate and mixtures thereof. These adjuvants are incorporated in the instant compositions
 15 of the present invention in amounts which do not substantially adversely affect the desired properties and characteristics and are suitably selected and used in conventional amounts.

For some purposes it may be desirable to include
 20 antibacterial agents in the compositions of the present invention. Typical antibacterial agents which may be used in amounts of 0.01% to 5%, preferably 0.05% to 1.0%, by weight of the dentifrice composition include:

N¹-4 (chlorobenzyl-N⁵-(2,4-dichlorobenzyl)
 25 biguanide;
 p-chlorophenyl biguanide;
 4-chlorobenz hydryl biguanide;
 4-chlorobenz hydrylguanylurea;
 N-3-lauroxypropyl-N⁵-p-chlorobenzylbiguanide;
 30 1,6-di-p-chlorobenzylbiguanide;

1-(lauryldimethylammonium)-8-(p-chlorobenzyl-
dimethyl-ammonium) octane dichloride;

5,6-dichloro-2-guanidinobenzimidazole;

N¹-p-chlorophenyl-N⁵-laurylbiguanide;

5 5-amino-1,3-bis (2-ethylhexyl)-5-methylhexa-
hydropyrimidine;

and their non-toxic acid addition salts.

Any suitable flavouring or sweetening materials
may be employed in formulating a flavour for the
10 compositions of the present invention. Examples of
suitable flavouring constituents include the flavouring
oils, e.g. oils of spearmint, peppermint, wintergreen,
sassafras, clove, sage, eucalyptus, marjoram, cinnamon,
lemon and orange as well as methylsalicylate. Suitable
15 sweetening agents include sucrose, lactose, maltose,
sorbitol, sodium cyclamate, sodium saccharine
dipeptides of U.S. Patent No.3,939,261 and oxathiazin
salts of U.S. Patent No. 3,932,606. Suitable flavour
and sweetening agent may together comprise from 0.01 to
20 5% of the composition.

The dentifrices should have a pH practicable
for use. A pH range of 3 to 10.5 is particularly
desirable. The reference to the pH is meant to be
the pH determination directly on the dentifrice. If
25 desired, materials such as benzoic or citric acid may
be added to adjust the pH to, say 4 to 8.5.

The invention may be put into practice in various
ways and a number of specific embodiments will be des-
cribed by way of example to illustrate the invention
30 with reference to the accompanying examples. All

amounts of the various ingredients are by weight unless otherwise specified.

EXAMPLES 1A to 1I

Example 1A

5 The following dentifrice is prepared by conventional room temperature cold process and filled into the mechanically operated dispenser described in British Patent Publication 2 070 695A.

		<u>Parts</u>
10	Sorbitol (70% aqueous solution)	23.000
	Sodium Saccharin	0.170
	i-carrageenan (Genuvisco 0319)	0.500
	Sodium Carboxymethyl cellulose (7MFD)	0.500
	Methyl p-hydroxybenzoate	0.080
15	Allantoin	0.150
	Sodium Fluoride	0.100
	Sodium Monofluorophosphate	0.760
	Pyridyl Carbinol	0.100
	Alpha alumina trihydrate (Alcoa	
20	(Registered Trade Mark) C-333)	51.000
	Benzoic Acid	0.140
	Sodium Lauryl Sulphate	1.667
	Titanium Dioxide	0.500
	Flavour	1.200
25	Deionised water	<u>20.133</u>
		100.000

The dentifrice fills efficiently into the dispenser and does not undergo leakage during storage at 43°C for over 1 month. It extrudes well from the dispenser after 1 month storage at 43°C.

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Similar satisfactory results are attained when the dentifrice is modified to contain 0.55 parts of each of i-carrageenan and sodium carboxymethyl cellulose in one case (Example 1B) and 0.45 parts of each of i-carrageenan and sodium carboxymethyl cellulose in a second case (Example 1C), with corresponding adjustment of the water contents to keep the total parts of the composition the same. Indeed absence of leakage and good extrudibility after storage at 43°C for 3 months are observed with these dentifrices.

Again satisfactory results are observed when each of the three dentifrices described above are modified to contain 50.000 parts of alpha-alumina trihydrate in one set of these dentifrices (Examples 1D, E and F) and 52.000 parts of alpha-alumina trihydrate in the second set of three dentifrices (Examples 1G, H and I), with corresponding adjustment of the weight of water present.

EXAMPLES 2A to 2D

Example 2A

The following dentifrice is prepared by cold process and filled into the same dispenser as in Example 1.

	<u>Parts</u>
25 Sorbitol (70% aqueous solution)	23.000
Sodium Saccharin	0.170
i-carrageenan (Genuvisco 0819)	0.666
Sodium carboxymethyl cellulose (7MFD)	0.334
Methyl p-hydroxybenzoate	0.080
30 Allantoin	0.150
Sodium Fluoride	0.100

	<u>Parts</u>
Sodium Monofluorophosphate	0.760
Pyridyl carbinol	0.100
Alpha alumina trihydrate (Alcoa C-333)	51.500
Benzoic Acid	0.140
5. Sodium lauryl sulphate	1.667
Titanium Dioxide	0.500
Flavour	1.200
Deionised water	19.633

10. The dentifrice fills efficiently into the dispenser and does not undergo leakage and extrudes well from the dispenser after storage for 1 month at 43°C.

Similar desirable effects are attained when the dentifrices of the examples are filled into a pressure differential dispenser.

15. When sodium carboxymethyl cellulose is used as the only gelling agent (Example 2B) poor extrusion occurs. When hydroxyethyl cellulose is used as the only gelling agent (Example 2C) leakage occurs.
20. However, when hydroxyethyl cellulose is mixed with i-carrageenan (Example 2D) desirable filling and extrusion is attained.

EXAMPLES 3A to 3C

Example 3A

25. The following dentifrice is prepared by conventional room temperature cold process and filled into the mechanically operated dispenser in British Patent Publication 2 070 695A.

		<u>Parts</u>
	Sorbitol (70% aqueous solution)	23.000
	Sodium Saccharin	0.170
	i-carrageenan (Genuvisco 0819	0.500
	Xanthan (Keltrol (Registered Trade Mark))	0.500
5	Methyl p-hydroxybenzoate	0.080
	Allantoin	0.150
	Sodium Fluoride	0.100
	Sodium Monofluorophosphate	0.760
	Pyridyl Carbinol	0.100
10	Alpha alumina trihydrate (Alcoa C-333)	51.000
	Benzoic Acid	0.140
	Sodium Lauryl Sulphate	1.667
	Titanium Dioxide	0.500
	Flavour	1.200
15	Deionised water	<u>20.133</u>

The dentifrice fills efficiently into the dispenser and does not undergo leakage during storage at 43°C for over 1 month. It extrudes well from the dispenser after 1 month storage at 43°C.

Similarly satisfactory results are attained when the dentifrice is modified to contain 0.55 parts of each of i-carrageenan and xanthan in one case (Example 3B) and 0.45 parts of each of i-carrageenan and xanthan in a second case (Example 3C), with corresponding adjustment of the weight of water present, to keep the total parts of the composition the same.

- Similar desirable results are attained with 50.000 parts of alpha-alumina trihydrate. Each variant also provides similar results with a gel system containing (a) 0.825 parts of i-carrageenan and 0.275 parts of xanthan, and (b) 0.675 parts of i-carrageenan and 0.225 parts of xanthan. Similar desirable effects are attained when the dentifrices of the examples are filled into a pressure differential dispenser.
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- It will be apparent to those skilled in the art that further modifications of the examples illustrative of the invention, may be made thereto.
- 10.

CLAIMS

1. A dentifrice comprising 20-80% by weight of
an aqueous humectant vehicle, 0.1-5% by weight of
5 gelling agent mixture, 20-75% by weight of
alpha-alumina trihydrate polishing agent, sodium
fluoride and sodium monofluorophosphate in amount to
provide 300 to 10000 ppm by weight of fluorine,
0.05-0.5% by weight of allantoin desensitising agent
10 and 0.05-0.5% by weight of pyridyl carbinol vasodilator
agent, wherein the said gelling agent mixture is a
mixture either of a cellulosic gelling agent or xanthan
with i-carrageenan in a weight ratio of cellulosic
gelling agent or xanthan to i-carrageenan of 5:1 to
15 1:5, subject to the proviso that the total of all the
ingredients is 100% by weight.

2. A dentifrice as claimed in Claim 1, in which
the weight ratio of the said cellulosic gelling agent
20 or of the said xanthan to the said i-carrageenan is 3:1
to 1:3.

3. A dentifrice as claimed in Claim 1 or Claim
2, in which the gelling agent mixture is present in
25 amount of 0.2-3% by weight.

4. A dentifrice as claimed in Claim 1, 2 or 3,
in which the said cellulosic gelling agent is present
and is sodium carboxymethyl cellulose.



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5. A dentifrice as claimed in Claim 1, 2 or 3, in which the said xanthan is present.

5 6. A dentifrice as claimed in any one of Claims 1 to 5, in which the weight ratio of the said cellulosic gelling agent or of the said xanthan to the said i-carrageenan is about 1:1.

10 7. A dentifrice as claimed in any one of Claims 1 to 6, in which the said sodium fluoride and the said sodium monofluorophosphate are present in amount to provide 750-2000 ppm by weight of fluorine and 30-40% by weight of the fluorine is provided by the said sodium fluoride.

15 8. A dentifrice as claimed in anyone of Claims 1 to 7 which in addition contains an anti bacterial agent in an amount up to 5% by weight.

20 9. A dentifrice as claimed in any one of Claims 1 to 8 which in addition contains flavour in an amount up to 5% by weight.

25 10. A dentifrice as claimed in any one of Claims 1 to 9 which in addition contains sodium lauryl sulphate in an amount of 1.7% by weight.

30 11. A dentifrice as claimed in Claim 1, substantially as specifically described herein with reference to any one of Examples 1A to 1I, 2A, 2D, 3A to 3C.



12. A dentifrice as claimed in any one of Claims 1 to 11, in which the said dentifrice is contained in a mechanically operated dispenser.

5 13. A dentifrice as claimed in any one of Claims 1 to 11, in which the dentifrice is contained in a pressure differential dispenser.

10 14. A process for preparing a dispenser containing a dentifrice as claimed in any one of Claims 1 to 11 which comprises extruding into a mechanically operated dispenser an amount of the said dentifrice through a nozzle to fill the dispenser which is open at its bottom and which contains a central rod, sliding
15 into place around the said central rod a piston having a diameter corresponding to the inner diameter of the dispenser and a central hole to receive the said central rod and then sealing the said dispenser with a bottom disc.

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