

- (21) Application No. 35143/71 (22) Filed 27 July 1971 (19)
 (31) Convention Application No. 61486 (32) Filed 5 Aug. 1970 in
 (33) United States of America (US)
 (44) Complete Specification published 3 Jan. 1974
 (51) International Classification B65D 35/08 79/00
 (52) Index at acceptance
 B8C 24B3 28 29F



(54) DENTIFRICE PACKAGE

(71) We, COLGATE-PALMOLIVE COMPANY, a Corporation organised and existing under the Laws of the State of Delaware, United States of America, of 300 Park Avenue, New York, New York 10022, United States of America, 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 package of a paste or gel dentifrice material in a tube.

According to the invention a dentifrice package comprises a clear transparent squeezable tube of a thermoplastic package material which is a nitrile-rubber modified acrylonitrile-methyl acrylate copolymer or a methacrylonitrile-styrene copolymer, the tube having therein a dentifrice which includes a flavour and is a homogeneous clear gel or is a paste or gel which has therein particles, speckles, stripes or bubbles which contrast visually with the background paste or gel.

The invention enables the aesthetically appealing appearance of such a paste or gel dentifrice material to be observable even while the dentifrice is still in the tube. Also, it has been found that the said thermoplastic package materials, in addition to retaining their clarity in the presence of the paste or gel, do not cause appreciable loss of flavour of the packaged paste or gel, in contrast to the flavour loss which may occur if such a paste or gel is packaged in a tube of some other clear thermoplastic materials such as polyethylene.

Examples of suitable thermoplastic package materials are the "Barex" resins available from Vistron Corporation, a subsidiary of Standard Oil Company of Ohio, U.S.A. and "Lopac" resins available from Monsanto Company, U.S.A. possess excellent clarity (BAREX and LOPAC are Trade Marks). "Barex" resins are nitrile-rubber modified acrylonitrile-methyl acrylate copolymers. "Barex 210" is about 73-77 parts by weight acrylonitrile, and about 27-23 parts by weight methyl acrylate in the presence of about 8-10 parts by weight of a buta-

diene-acrylonitrile copolymer which is about 70% by weight derived from butadiene. "Lopac" resins appear as methacrylonitrile-styrene copolymers and have been named "Lopacs" 90, 110, 210, 310, and 410. "Lopac 90" appears as about 86% by weight of acrylonitrile and about 13% by weight of styrene.

These thermoplastic polymers besides being clear, are relatively light, typically having a specific gravity between about 0.9 and about 1.5 and are dimensionally stable and easily flexible to permit extrusion of paste or gel dentifrices contained in tubes formed thereof, by squeezing the tubes, over a wide temperature range, e.g. -20°C to +45°C.

The thermoplastic material may be extruded into the desired length and cylindrical shape of a dentifrice tube which typically has a thickness of about 0.01 to about 0.025 mm, preferably 0.01 to 0.018 mm. The neck of the desired thread diameter, neck length, finished snip height and orifice opening, and the shoulders of the desired length and angle from the tube diameter, are typically formed by injection moulding. The tube, still open at both ends, is then generally capped at the neck, filled with paste or gel and heat sealed at the bottom. Descriptive printing may also be placed on the tubes during the filling and sealing operations.

The dentifrice which fills the tube may be of known composition and will generally contain a polishing agent in a vehicle which is primarily a liquid-gel system. The liquid generally includes water and/or a humectant, such as glycerine, sorbitol, propylene glycol and polyethylene glycol 400 including suitable mixtures thereof, and a gelling or thickening agent such as carboxymethyl cellulose, Irish Moss, gum tragacanth, gum karaya, hydroxymethyl carboxyethyl cellulose, methyl cellulose, polyvinyl alcohol, polyvinyl pyrrolidone, gelatin starch, glucose or sucrose. Typically, the liquid comprises 20% to 90% by weight of the dentifrice and the gelling agent 0.25% to 10% by weight. These and all other percentages relating to components of the dentifrices are by weight of the dentifrice. In pastes which

are opaque, the liquid preferable includes 15% to 35% glycerine and 15% to 30% water. In gels which are transparent or translucent, the liquid preferably includes up to 20% water, 15% to 40% glycerine and up to 50% sorbitol.

Various polishing agents may be incorporated into the liquid-gel system, generally in an amount of 5% to 75% (preferably 5% to 50%) in a clear or translucent gel and 20% to 75% in an opaque paste. Typical polishing agents which may be employed are dicalcium phosphate, tricalcium phosphate, insoluble sodium metaphosphate, aluminium hydroxide, calcium carbonate, magnesium carbonate, calcium pyrophosphate, bentonite, silica including crystalline silica and colloidal silicic anhydride, and mixtures thereof. In a clear or translucent gel, colloidal silicic anhydride and alkali metal or alkaline earth metal aluminosilicates are preferably employed.

The liquid-gel system may also include minor amounts of additional ingredients, such as 0.05% to 10% of a surface active agent, 0.1% to 5% of an antibacterial agent, as well as preservatives, silicones, chlorophyll compounds, ammoniated materials such as urea and/or diammonium phosphate, and colour contrasting materials such as zinc oxide or titanium dioxide.

Typical surface active agents are alkyl sulphates such as sodium lauryl sulphate, higher aliphatic acyl amide of lower aliphatic amino carboxylates such as sodium N-lauroyl sarcosinate, water-soluble salts of fatty acid monoglyceride sulphates such as sodium coconut fatty acid monoglyceride monosulphate, and alkyl aryl sulphonates such as sodium dodecyl benzene sulphonate.

Typical bacteriostatic compounds include N¹ - (4-chloro-benzyl)-N⁵ - (2,4-dichlorobenzyl)biguanide and 1,6-(di-p-chloro-phenylbiguanide)hexane.

Fluorine-containing compounds such as sodium fluoride, stannous fluoride and sodium monofluorophosphate may also be present, typically 0.01% to 1% calculated as the water-soluble fluorine content of the compound.

The paste, or gel also includes a flavour, desirably 0.01% to 5% or even more of a sialogogue flavour or sweetener. Examples of suitable flavours are flavouring oils, such as oils of spearmint, peppermint, wintergreen, sassafras, clove, sage, eucalyptus, marjoram, cinnamon, lemon and orange. Sweeteners include methyl salicylate, sucrose, lactose, maltose, sorbitol, sodium cyclamate (where permitted) and saccharin may also be used. The sialogogue effect may also be modified with chloroform.

In order to enhance the visual appeal of the dentifrice in the clear tube, the dentifrice may have coloured stripes which may

include a high proportion of the bacteriostate or flavour in the dentifrice, or visible particles, such as an insoluble salt of carboxymethyl cellulose, mother of pearl flakes or other iridescent flakes such as mica flakes coated with titanium dioxide, hollow polyethylene particles having incorporated therein ingredients such as a polishing agent, e.g. zirconium silicate, or other materials which are visible in translucent or transparent dentifrices and can be satisfactorily used in a dentifrice.

During the manufacture of dentifrices, it has been conventional to remove entrained air from the product by deaeration under vacuum. With a transparent tube, and particularly with translucent or transparent gel dentifrices of suitable viscosity, dispersed, immobile air bubbles may enhance the visual attraction of the package and can, therefore, be permitted to remain. Furthermore, air can be at least partially removed and re-introduced as substantially globular or spheroidal bubbles of about 0.1 mm to about 8 mm, preferably 0.5 mm to 5 mm, in diameter, well distributed in the dentifrice to be packaged, at an average of at least one bubble per cubic centimetre. Such air bubbles may be placed in the dentifrice by stirring while introducing air. Instead of air, bubbles of another gas, such as nitrogen or carbon dioxide, can be introduced in non-toxic quantity. In particular, carbon dioxide can provide an effervescent appearance to the dentifrice.

The following Example illustrates the invention. All amounts are by weight.

EXAMPLE

Thermoplastic package material, namely "Barex 210" is extruded into cylindrical shape, injection moulded to form the tube neck and shoulder, capped at the neck, positioned with the open end up and filled with a desired amount of dentifrice paste or gel. Printing can be applied to the tube at this time. It is then closed at the bottom by clamping and heat sealing. The heat sealing makes the end soft, and it can then be again squeezed and air cooled.

Components	Parts
Glycerine	25
Sorbitol (70% aqueous solution)	47
Sodium carboxymethyl cellulose	0.35
Non-abrasive silica thickener — "Syloid 244" (SYLOID is a Trade Mark) manufactured by Grace, Davidson Chemical Co., U.S.A.	3
Colloidal silicic anhydride — "Syloid 74" manufactured by Grace, Davidson Chemical Co., U.S.A.	18
Sodium lauryl sulphate	2
	120
	125
	130

Components	Parts
Water	3.5
Sodium benzoate	0.5
Sodium saccharin	0.17
5 Flavour	1.1
Mother of pearl flakes	2

This iridescently speckled clear gel is easily squeezed from the plastic tube.

10 Instead of the mother of pearl flakes, a controlled amount of discrete, immobile air bubbles of an average diameter of about 1 mm, can be introduced into the gel.

15 The packaged tubes retain their clarity over a long period of time and the gel is easily seen inside the tube.

Similar tube clarity and good flavour retention are observed when the tube material is "Lopac 90". Other dentifrices, including the above clear gel from which the flakes are omitted, and gels and pastes containing visible agglomerates of polishing agent, speckles of aluminium carboxymethyl cellulose and flavour stripes, all can be easily and clearly seen in the plastic tubes.

25 It is also within the scope of this invention to impregnate the clear thermoplastic squeezable tube with particles such as iridescent mother of pearl flakes to increase the visual appeal of the package.

WHAT WE CLAIM IS:—

1. A dentifrice package comprising a clear transparent squeezable tube of a thermoplastic package material which is a nitrile-rubber modified acrylonitrile-methyl acrylate copolymer or a methacrylonitrile-styrene copolymer, the tube having therein a dentifrice which includes a flavour and is a homogeneous clear gel or is a paste or gel which has therein particles, speckles, stripes or bubbles which contrast visually with the background paste or gel.

2. A dentifrice package as claimed in Claim 1 wherein the thermoplastic package material is a nitrile-rubber modified acrylonitrile-methyl acrylate copolymer which contains 73 to 77 parts by weight of acrylonitrile and 27 to 23 parts by weight of methyl acrylate in the presence of 8 to 10 parts by weight of a butadiene-acrylonitrile copolymer which is about 70% by weight derived from butadiene.

3. A package as claimed in Claim 1 or Claim 2 wherein the dentifrice is a clear gel which includes visible iridescent speckles.

4. A dentifrice package substantially as described in the Example.

KILBURN & STRODE,
Chartered Patent Agents,
Agents for the Applicants.

Components	Parts
Water	3.5
Sodium benzoate	0.5
Sodium saccharin	0.17
5 Flavour	1.1
Mother of pearl flakes	2

This iridescently speckled clear gel is easily squeezed from the plastic tube.

10 Instead of the mother of pearl flakes, a controlled amount of discrete, immobile air bubbles of an average diameter of about 1 mm, can be introduced into the gel.

15 The packaged tubes retain their clarity over a long period of time and the gel is easily seen inside the tube.

20 Similar tube clarity and good flavour retention are observed when the tube material is "Lopac 90". Other dentifrices, including the above clear gel from which the flakes are omitted, and gels and pastes containing visible agglomerates of polishing agent, speckles of aluminium carboxymethyl cellulose and flavour stripes, all can be easily

25 and clearly seen in the plastic tubes.
It is also within the scope of this invention to impregnate the clear thermoplastic squeezable tube with particles such as iridescent mother of pearl flakes to increase
30 the visual appeal of the package.

WHAT WE CLAIM IS:—

1. A dentifrice package comprising a clear transparent squeezable tube of a thermoplastic package material which is a nitrile-rubber modified acrylonitrile-methyl acrylate copolymer or a methacrylonitrile-styrene copolymer, the tube having therein a dentifrice which includes a flavour and is a homogeneous clear gel or is a paste or gel which has therein particles, speckles, stripes or bubbles which contrast visually with the background paste or gel. 35 40

2. A dentifrice package as claimed in Claim 1 wherein the thermoplastic package material is a nitrile-rubber modified acrylonitrile-methyl acrylate copolymer which contains 73 to 77 parts by weight of acrylonitrile and 27 to 23 parts by weight of methyl acrylate in the presence of 8 to 10 parts by weight of a butadiene-acrylonitrile copolymer which is about 70% by weight derived from butadiene. 45 50

3. A package as claimed in Claim 1 or Claim 2 wherein the dentifrice is a clear gel which includes visible iridescent speckles. 55

4. A dentifrice package substantially as described in the Example.

KILBURN & STRODE,
Chartered Patent Agents,
Agents for the Applicants.



* C L M S *