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Codispensing of physically segregated dentifrices at consistant ratios

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(54) Title: CODISPENSING OF PHYSICALLY SEGREGATED DENTIFRICES AT CONSISTANT RATIOS

(57) Abstract

A method is disclosed for the coextrusion in controlled amounts of at least two dentifrice components stored and physically segregated in a multicompartmented collapsible dispenser provided with a partition which divides the interior volume of the container into separate compartments, the partition being moveable in response to a pressure differential developed thereacross upon the application of Compressive Force to the sidewalls, the individuals dentifrice components containing ingredients which interact when mixed, the dispenser sidewalls being formed of a resilient plastic material which is deflected upon the application thereto of a Deflective Force of about 1.0 or greater pounds, the dentifrice compnents being formulated to be equally extrudible at substantially equivalent compressive forces. When the dispenser sidewalls are compressed, there is extruded a single-banded unmixed multilayer stream of the components containing the reactive ingredients at ratios predetermined to provide optimum levels for interaction between the reacted ingredients when the extruded components are mixed in the oral cavity.

CODISPENSING OF PHYSICALLY SEGREGATED DENTIFRICES AT CONSISTANT RATIOS

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention is directed to a packaging system for storing and simultaneously dispensing physically separated components of a multicomponent dentifrice at constant predetermined ingredient ratios in a multilayer unmixed relationship.

2. Prior Art

Multicomponent dentifrice compositions are known to the art wherein the individual components containing reactive ingredients are physically segregated during storage and are simultaneously dispensed as viscous paste or liquid materials which interact when mixed with each other, as during brushing, the components being dispensed at specific predetermined ingredient ratios for optimum efficacy and performance.

For example, it has long been known to include fluoride releasing compounds in dentifrices as anticaries agents, and it has been established that these compounds are effective to reduce the incidence of dental caries. The effectiveness of the dispensed fluoride is dependent upon the amount of fluoride ion which is available for deposition on the dental tissue being treated. It is, therefore, desirable to formulate dentifrice compositions which provide maximum fluoride ion availability in brushing solutions formed using the dentifrice.

One effective way of depositing fluoride on teeth is to use a two-component dentifrice composition comprised of a calcium containing component and the other a fluoride containing component to precipitate calcium fluoride on teeth.

For example, US 5,045,305 teaches a two component dentifrice for fluoridating teeth in which one component contains $CaCl_2$ and the other contains fluoride ions in the

form of NaF, the separate components being admixed immediately prior to introduction in the mouth to effect precipitation of CaF_2.

US Patent No. 5,145,668 discloses a method of fluoridating teeth wherein there is mixed during toothbrushing a first component comprising a soluble calcium salt such as $CaCl_2$ and a second component containing a hydrolyzable complex fluoride compound such as sodium fluorosilicate (Na_2SiF_6) the mixing of the components resulting in hydrolysis of the complex fluoride compound and precipitation of calcium fluoride and its deposition on tooth surfaces.

US 5,476,647 discloses a two-component fluoride deposition system wherein the first component of the system contains a soluble calcium source and a soluble Ca-complexing anion such as ethylene diaminetetraacetic acid, the calcium being partially bound to the Ca-complex agent. The second component contains a fluoride salt such as sodium fluoride or stannous fluoride. When the two components are combined, precipitation of calcium fluoride (CaF_2) removes free Ca^{2+} from the solution releasing of additional free Ca^{2+} from the calcium complexing agent, which, in turn, causes additional CaF_2 to precipitate.

In U.S. 5,785,956 there is disclosed a dentrifice for fluoridating teeth utlizing two separate physically segregated semi-solid dentifrice components which contain fluoride salt and calcium ion containing abrasive ingredients which are reactive when mixed together upon application to teeth, the first component being an aqueous, semi-solid dentifrice composition containing a fluoride ion releasing salt such as an alkali metal salt such as sodium fluoride in a vehicle in which the ingredients thereof are non-reactive with the fluoride salt, and the second component is comprised of a vehicle containing a calcium ion containing abrasive such as hydrated dicalcium phosphate wherein the individual dentifrice components are substantially rheologically equivalent and are simultaneously extruded from a dual compartmented container. When the ingredients in the dispensed components are at the proper reactant ratio maximum fluoride availability is provided as precipitated calcium fluoride upon mixing of the dentifrice components during application to the teeth as by brushing.

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The most convenient and least cost way to dispense physically separated components of multi-component dentifrices is from a collapsible, compartmented plastic tube. Dual compartmented collapsible tubes for the simultaneous coextrusion of two

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physically separated dentifrice components are known to the art whereby compression of the tube, as by squeezing, dispenses a single banded multilayer ribbon product. The bodies of these dual compartmented tubes are typically sealed at one end and are manufactured from plastic sheet. The structure of the dual compartmented tube further includes a partition within the tube body for defining separate compartments therein and a relatively rigid neck portion is provided at the unsealed end having a discharge opening extending therethrough, the outer peripheral surface of the neck portion being threaded or otherwise constructed to enable a cap to be threaded or otherwise attached thereon to seal the tube. Examples of such dual compartmented tube structures for dispensing multicomponent dentifrices are disclosed in U.S. Patents 4,481,757 and 4,687,663.

U.S. 4,487,757 discloses a dual compartmented collapsible tube separated into two compartments by a partition which extends to the dispensing nozzle which physically segregates and dispenses a dual component toothpaste containing reactive ingredients, wherein one component contains an alkali metal bicarbonate salt (e.g., $NaHCO_3$) and the other contains an acid or acid salt (e.g., citric acid) which is reactive with the bicarbonate salt to produce effervescence (carbon dioxide) upon simultaneous dispensing of unmixed layers of both components and intimate mixing thereof during toothbrushing.

U.S. 4,687,663 discloses a dual compartmented tube in which the dual component dentifrice dispensed is composed of a first hydrogen peroxide containing gel and a sodium bicarbonate containing second component. The tube is separated into two compartments by a partition extending into the dispensing nozzle.

A disadvantage experienced by the prior art with two component dispensing systems such as are disclosed in the above discussed patents is that efforts to utilize such systems using semi-solid, extrudable oral care products such as toothpastes and gels containing reactive ingredients which require constant predetermined ingredient ratios to achieve optimum performance is that unequal dispensing of the dentifrice components from plastic compartmented tubes occurs so that optimum interaction of the reacted ingredients when the two components are mixed during brushing of the teeth, is not achieved. For example, U.S. 5,137,178 discloses (col. 1, lines 29-39) that a common problem of the known art in dispensing dual component reactive products is the inability to control the relative flow of each component from its respective compartment in a dual compartmented container, there being a particular problem in extruding equal volumes of the two components. U.S. 5,020,694 (col. 1, line 64 to col. 3, line 3) discloses that the dual compartmented tubes of the prior art as represented by previously discussed U.S.

4,487,757 and U.S. 4,687,663 suffer from the disadvantage that the tubes tend to dispense dual component dentifrice materials in uncontrolled, varying proportions even though the rheologies of the components are similar.

Unequal dispensing of the components of a multi-component dentifrice causes a variation in the component layers in the multilayer dentifrice portion which is next dispensed, so that although the components which are next dispensed are dispensed synchronously, the ingredients in the dispensed components are not matched at the correct reactant ratios for optimum interaction of the reactive ingredients. In the case of multicomponent dentifrices of the type disclosed in US 5,785,956 used for the fluoridation of teeth, maximum fluoride availability as precipitated calcium fluoride is not achieved when the unequally dispensed components are mixed during brushing.

U.S. Patents 5,020,694 and U.S. 5,038,963 describe rigid piston type multicompartmented dispensing containers for simultaneous coextrusion of two or more dentifrice components in a predetermined proportion. These rigid containers have the advantage of control over the coextrusion process. However a considerable amount of plastic material is involved in their construction. For environmental and cost reasons, packaging with less plastic material is commercially desirable. Therefore, there is a need in the art for a non-rigid plastic compartmented container or tube for storing and simultaneously dispensing physically separated dentifrice components containing reactive ingredients at constant predetermined ingredient ratios whereby optimum interaction between reactive ingredients is obtained, such container requiring considerably less plastic material for its manufacture.

SUMMARY OF THE INVENTION

In accordance with the present invention there is provided a means for the constant codispensing in predetermined proportions of physically separated dentifrice components containing different reactive ingredients from a multicompartmented, non-rigid, container having collapsible sidewalls, the container including (1) a collapsible body portion formed from a plastic web material which deforms under a deflective force of about

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0.45 kg or greater applied thereto and (2) a moveable partition within the container body defining separate compartments, the partition being a membrane which has a thickness less than that of the body sidewalls which deforms under a deflective force of less than 0.23 kg and is displaced in response to a pressure differential developed thereacross upon compression of the container sidewalls, the displacement of the membrane substantially vitiating the pressure differential and equalizing the compressive forces exerted on the dentifrice components housed in the container compartments, whereby substantially constant codispensing of the components in predetermined proportions is attained when the components are formulated such that the compression forces required to extrude the individual dentifrice components are substantially equivalent.

Throughout this specification the word "comprise", or variations such as "comprises" or "comprising", will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The Dentifrice Components

The term "substantially equivalent Compressive Force" as used herein means that the individual components of the multicomponent dentifrice of the present invention are formulated so that the force required to actuate the flow and extrusion of an individually housed dentifrice component from the container outlet does not differ by more than 20% from any other separately housed component so that correctly proportioned interaction between the different reactive ingredients of the components is achieved when the components are ultimately mixed together.

It has been determined that individual dentifrice components when they contain different reactive ingredients encounter different surface forces with respect to the plastic sidewalls of the collapsible, non-rigid container in which they are housed as they are extruded from the container compartments

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and therefore to provide for simultaneous coextrusion of the individual components wherein the reactive ingredients contained therein are in a predetermined proportion, the vehicles used to prepare the individual dentifrice components must be adjusted to account for the different surface forces encountered by the dentifrice components being extruded so that the compressive force necessary for controlled coextrusion is substantially equivalent.

It has been further determined that substantially constant codispensing of dentifrice components containing different reactive ingredients is achievable with the codispensing system of the present invention when the individual dentifrice components are formulated to be extrudable under a Compressive Force of about 0.23 to about 9 kg and preferably about 0.45 to about 4.5 kgs.

The term "Compressive Force" measured in kilograms (kg), as used herein, and in the appended claims, means the force required to extrude 1-2 grams of a dentifrice

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component housed in a stainless steel container, a distance of 5 millimeters (mm) through an aperture opening of 3.8mm diameter at a temperature of 23°C at a fixed rate of 150 millimeters/minute (mm/min). To measure such a compressive force, the dentifrice component is placed in a stainless steel container having a 2.0 centimeter (cm) inside diameter. The bottom of the stainless steel container is equipped with an orifice which is 20mm in length and has an inside orifice diameter of 0.38cm. A frictionless teflon piston with an outside diameter which is equal to the inside diameter of the stainless steel container is placed on top of the dentifrice. An Instron® compression tester is operated to measure the force required to move the teflon piston down a 5mm distance at 150 mm/min. The Compressive Force required for extrusion is obtained at the inflection point (change in slope) in a force vs. distance curve; the force applied by the piston causing the dentifrice component to flow increasing linearly until there is a change in the slope indicating the initiation of dentifrice component extrusion. To obtain equal dispensing of the separately housed dentifrice components it is critical to the practice of the present invention, that the compression force measured at the inflection point for each component be within $\pm$20% of the other.

The compressive force required to effect extrusion of the dentifrice components of the present invention may be adjusted by the selection and concentration of the ingredients of the dentifrice vehicle in which the reactive ingredients are incorporated.

The dentifrice vehicle used in the practice of the invention is adjusted to impart to the dentifrice component the pasty consistency, body and non-tacky nature which is characteristic of conventional toothpastes or gels. The vehicle is non-reactive with the reactive ingredients of the dentifrice and includes water, a suitable humectant such as glycerin, sorbitol, propylene glycol, polyethylene glycol, or any suitable mixture thereof and a thickening agent.

The humectant typically comprises about 10 to about 70% by weight of the dentifrice component and preferably about 20 to about 50% by weight of the dentifrice component Water may be included in the vehicle at a concentration of about 6 to about 70% by weight of the component and preferably about 15 to about 40% by weight.

Thickening agents incorporated in the dentifrice components of the present invention include natural and synthetic gums and colloids examples of which include iota carrageenan, kappa carrageenan, lamda carrageenan and mixtures thereof, xanthan gum, carboxymethyl cellulose, sodium carboxymethyl cellulose, starch, polyvinylpyrrolidone,

hydroxyethylpropylcellulose, hydroxybutyl methyl cellulose, hydroxypropyl methyl cellulose, hydroxyethyl cellulose, laponites and magnesium aluminum silicates. Preferred thickening agents are thickeners which impart elastic structure to the dentifrice components so that progressive, variable thickening of the dentifrice component on storage is avoided. When such thickeners are used in the preparation of the dentifrice components used in the practice of the present invention the compressive force required for extrusion of the dentifrice component does not substantially change during storage. Examples of thickeners which impart elastic structure include the different types of carrageenan, laponites, magnesium aluminum silicates available commercially under the tradename Veegum and polyoxyethylene-polyoxypropylene block copolymers available under the trademark "Pluronic".

A thickener which imparts the desired elastic structure to dentifrice components which is most is preferred in the practice of the present invention is a cellulose gel formed from a dried, spray dried or bulk dried, co-processed, mixture of a microcrystalline cellulose and a cellulose gum such as carboxymethylcellulose, xanthan gum or sodium alginate. An example of such cellulose gels are those sold by the FMC Corporation under the tradename Avicel which generally contain about 80 to about 90% by weight microcrystalline cellulose and about 10 to about 20% by weight cellulose gum. The particle size range of the gel is from submicron, that is, less than 0.2 micron to about 100 microns, and preferably about 0.2 to about 20 microns. A cellulose gel particularly preferred for use in the practice of the present invention is Avicel RC-591-F which is a spray dried cellullose gel having the following composition and properties:

% Microcrystalline Cellulose	88
% Colloidal (0.2 micron)	70
% Carboxymethyl cellulose	12
Initial Viscosity*	39-175 at 1.2%
Set Up Viscosity**	1250 cps at 1.2%

* Initial Viscosity: 120 secs. using a Brookfield® RVT Viscometer #1 spindle at 20 rpms (revolutions per minute).

** Set-up Viscosity: 24 hours using a Brookfield® RVT Viscometer #3 spindle at 20 rpms.

Thickening agents such as Avicel RC-591-F may be incorporated in the dentifrice components of the present invention at a concentration of about 0.05 to about 2% by weight and preferably about 0.1 to about 1.5% by weight.

Surfactants are used in the preparation of dentifrice components of the present invention to aid in the thorough dispersion of the dentifrice components throughout the oral cavity when applied thereto as well as to improve the cosmetic acceptability and detersive and foaming properties of the combined components. The surfactant is included in the dentifrice vehicle of the individual components of the present invention at a concentration of about 0.5 to about 3.0% by weight and preferably about 1.0 to about 2.0% by weight.

Among the surfactants useful in the practice of the present invention are salts of the higher alkyl sulfates, such as sodium lauryl sulfate or other suitable alkyl sulfate having 8 to 18 carbon atoms in the alkyl group; sodium lauryl sulfoacetate, salts of sulfonated monoglycerides of higher fatty acids, such as sodium coconut monoglyceride sulfonate or other suitable sulfonated monoglycerides of a fatty acids of 10 to 18 carbon atoms; salts of amides of higher fatty acid, e.g., 12 to 16 carbon atom acids, with lower aliphatic amino acids, such as sodium-N-methyl-N-palmitoyl tauride, sodium N-lauroyl-, N-myristoyl- and N-palmitoyl sarcosinates; salts of the esters of such fatty acids with isotonic acid or with glycerol monosulfate, such as the sodium salt of monosulfated monoglyceride of hydrogenated coconut oil fatty acids; salts of olefin sulfonates, e.g. alkene sulfonates or alkene sulfonates or mixtures thereof having 12 to 16 carbon atoms in the carbon chain of the molecule; and soaps of higher fatty acids, such as those of 12 to 18 carbon atoms, e.g., coconut fatty acids. The cation of the salt may be sodium, potassium or mono-, di or triethanol amine.

The practice of the present invention applies generally to dentifrice components which must be physically segregated from the other when each individually contains an ingredient reactive with the other. For example, peroxide compounds are very reactive ingredients and need to be separated from many dentifrice ingredients such as flavor compounds, vitamins (A, C, E), antibacterial agents such as triclosan, and polyphosphates such as sodium tripolyphosphate. Cationic compounds such like stannous ion, chlorhexidine digluconate, and cetyl pyridinium chloride also must be separated from anionic components of dentifrices such as tartar control agents (sodium tripolyphosphate), calcium and silica abrasives, and the like.

By way of more specific example, when it is desired to fluoridate teeth by precipitation of calcium fluoride, the first component of the dual component dentifrice composition contains a water soluble, fluoride releasable salt such as an alkali metal

fluoride such as NaF, KF, sodium monofluorophosphate or SnF_2. The preferred fluoride salt for the purposes of the present invention is NaF. The fluoride salt is incorporated in the first component of the dentifrice composition at a concentration of about 0.1 to about 1% by weight, and preferably at about 0.25 to about 0.5% by weight. At these preferred concentrations, about 750 ppm to about 1500 ppm, fluoride ion will be available to teeth when the combined first and second components of the dentifrice composition are admixed when applied to the teeth.

Other specific examples of reactive ingredients which may be incorporated in the first dentifrice component include peroxide compounds such as hydrogen peroxide and calcium peroxide or organic acidic ingredients such as citric acid or salts thereof which may be included in the first dentifrice component at a concentration of about 0.5 to about 5% by weight of the component.

The second component of the dentifrice composition of the present invention includes a reactive polishing or abrasive ingredient such as dicalcium phosphate or an alkali metal bicarbonate salt. When the second component of the dentifrice composition contains a hydrated dicalcium phosphate abrasive or a dicalcium orthophosphate dihydrate, the abrasive is incorporated in the second component of the dentifrice composition of the present invention at a concentration of about 1 to about 60% by weight and preferably at about 20 to about 50% by weight of the second component. When the abrasive is an alkali metal bicarbonate salt such as sodium bicarbonate, the bicarbonate salt is incorporated in the second component at a concentration of about 5 to about 25% by weight.

Other specific examples of two component dentifrice compositions which may be used in the practice of the present invention include dentifrice components containing ingredients which are normally reactive and incompatible with each other such as calcium containing abrasives and polycarboxylated polymers (e.g., Gantrez) or potassium nitrate.

The reactive abrasive is contained in a vehicle formulated to have a composition similar to the vehicle of the first dentifrice component, so that the two components will be of similar physical characteristics, which will permit them to be more readily formulated to be coextrudable at substantially equivalent compressive forces. For example, in order that the compressive force is substantially equivalent to extrude both components at a precise predetermined ratio to effect optimum precipitation of calcium fluoride, the vehicle composition of the second component, specifically the humectant content, is adjusted to

accommodate the difference in component ingredients. Thus, in the exemplary two component dentifrice in which one component contains fluoride and the other calcium abrasive, the humectant content of the second component is adjusted to accommodate the inclusion of the dicalcium phosphate abrasive. The abrasive is included in the second dentifrice component at a concentration of about 10 to about 30% by weight and preferably at a concentration of about 15 to about 25% by weight. At these abrasive levels, the humectant concentration ranges from about 15 to about 70% by weight and preferably about 20 to about 40% by weight.

Similar adjustments in sodium bicarbonate concentrations in the second dentifrice component of the multicomponent dentifrice used in the present invention can be made so that the compressive forces required to extrude both components are substantially equivalent to provide component dispensing at a predetermined ratio to effect optimum reaction and effervesce between the bicarbonate ingredient in the second component and the organic acid or peroxide ingredient in the first component.

A striped dentifrice product is obtained in accordance with the practice of the present invention wherein colorants of contrasting colors are incorporated in each of the dentifrice components used in the practice of the present invention, the colorants being pharmacologically and physiologically non-toxic when used in the suggested amounts. Colorants used in the practice of the present invention include both pigments and dyes.

Pigments used in the practice of the present invention include non-toxic, water insoluble inorganic pigments such as titanium dioxide and chromium oxide greens, ultramarine blues and pinks and ferric oxides as well as water insoluble dye lakes prepared by extending calcium or aluminum salts of FD&C dyes on alumina such as FD&C Green #1 lake, FD&C Blue #2 lake, FD&C R&D #30 lake and FD&C # Yellow 15 lake. The pigments have a particle size in the range of 5-1000 microns, preferably 250-500 microns, and are present at a concentration of 0.5 to 3% by weight.

The dyes used in the practice of the present invention are distributed uniformly throughout the dentifrice component and are generally food color additives presently certified under the Food Drug & Cosmetic Act for use in food and ingested drugs, including dyes such as FD&C Red No. 3 (sodium salt of tetraiodofluorescein), FD&C Yellow No. 5 (sodium salt of 4-p-sulfophenylazo-1-p-sulfophenyl-5-hydroxypyrazole-3 carboxylic acid), FD&C Yellow No. 6 (sodium salt of p-sulfophenylazo-B-naphtol-6-monosulfonate), FD&C Green No. 3 (disodium salt of 4-{[4-(N-ethyl-p-

sulfobenzylamino)-phenyl]-(4- hydroxy-2-sulfoniumphenyl)-methylene}-[1-(N-ethyl-N-p-sulfobenzyl)-Æ-3,5-cyclohexadienimine], FD&C Blue No. 1 (disodium salt of dibenzyldiethyl-diaminotriphenylcarbinol trisulfonic acid anhydrite), FD&C Blue No. 2(sodium salt of disulfonic acid of indigotin) and mixtures thereof in various proportions. The concentration of the dye for the most effective result in the present invention is present in the dentifrice composition in an amount from about 0.0005 percent to about 2 percent by weight.

It is preferred that the colorant included in one of the dentifrice components be a pigment such as TiO_2 and that colorant distributed throughout the body of the other dentifrice component be a dye and the dye be of a different color than the pigment included in the first dentifrice component.

Any suitable flavoring or sweetening material may also be employed. Examples of suitable flavoring constituents are flavoring oils, e.g., oils of spearmint, peppermint, wintergreen, sassafras, clove, sage, eucalyptus, marjoram, cinnamon, lemon, and orange, and methyl salicylate. Suitable sweetening agents include sucrose, lactose, maltose, sorbitol, sodium cyclamate, perillartine, and sodium saccharin. Suitably, flavor and sweetening agents may together comprise from 0.01% to 5% or more of the preparations.

Various other materials may be incorporated into the dentifrice components of this invention. Non-limiting examples thereof include preservatives, silicones and chlorophyll compounds, antibacterial agents such as chlorohexidene, halogenated diphenyl ethers such as triclosan, desensitizing agents such as potassium nitrate and potassium citrate and mixtures thereof. These adjuvants are incorporated in the dentifrice components in amounts which do not substantially adversely affect the properties and characteristics desired, and are selected and used in proper amounts, depending upon the particular type of dentifrice component involved.

To prepare the individual dentifrice components of the present invention, the humectants e.g. glycerin, polyethylene glycol ingredients and sweetener are dispersed in a conventional mixer until the mixture becomes a homogeneous gel phase. Into the gel phase are added the fluoride salt or dicalcium phosphate abrasive. These ingredients are mixed until a homogeneous phase is obtained. Thereafter the thickener, flavor and surfactant ingredients are added and the ingredients mixed at high speed under vacuum of about 20-100 mm Hg. The resultant product is a homogeneous, semi-solid, extrudable paste product.

The Dispensing Container

The material from which the body side walls of the multicompartmented container used in the practice of the present invention is manufactured is a flexible, resilient plastic material which is deflected when a force is applied to the web. In the practice of the present invention plastic web materials which deflect upon the application of a Deflective Force of about 0.45 to about 1.35 kg applied thereto have been found to be particularly suitable for codispensing dentifrice components from collapsible compartmented tubes in constant predetermined proportions in accordance with the practice of the present invention.

The term "Deflective Force" as used herein and in the appended claims means the maximum force expressed in kilograms required to deflect a plastic web bent in the form of an inverted U by a shaped adapter fitted to a compression table such as an Instron® Tensile Testing Machine, the force being applied axially downward on the arcuate section of the U-shaped web at a rate of 30 cm per minute.

The adapter installed on the Instron® Machine is 13.75 cm high and consists of a 0.64 cm thick stainless steel block, 2.5 cm square, with a 0.32 cm diameter stainless steel wire curving downwardly to an open rectangular section 12.5 cm wide and 6.25 cm high. The adapter is fitted into the jaws of the Instron® Machine and is moved downwardly to contact and deflect the surface web being tested.

The plastic web being tested is held in a specimen holder consisting of a stainless steel base 0.32 cm in thickness having a slot 2.5 cm wide, 10 cm long and 5 cm high. A lower mount for this base, 2.5 cm in length, mounts the base to the work platform of the Instron® Machine. A specimen brace fits into the base to hold the plastic web in the base, the brace consisting of a channel 10 cm in length, 2.47 cm wide and 5 cm high having a wall thickness of 1.56 cm. The specimen brace holds the web in the base in an inverted U shape.

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When making a measurement of Deflective Force, six plastic web specimens cut in the machine direction and six plastic web specimens cut in the cross direction, each specimen being 10 cm X 10 cm, are tested. Each specimen being tested is placed in the specimen holder and held in place by the brace so that it forms an inverted U shape. No specimen sample is reused. The specimen holder with the sample is placed in the Instron® Machine and the adapter lowered to just above the specimen and then lowered at the rate of 30 cm per minute to effect a web deflection of 0.875 cm. The force in kilograms to deflect the web in this manner is recorded as the Deflection Force.

The plastic sheet material from which the body sidewalls are manufactured may be made of any suitable plastic material such as polyethylene (both low and high density) polypropylene, ethylene and propylene copolymers and polyethylene terephthalate. The plastic sheet material may have a laminate structure wherein a gas barrier material such as ethylene vinyl alcohol, nylon or polyvinylidene chloride is sandwiched between layers of polyethylene or polypropylene or copolymers thereof. The gas barrier materials prevent the loss of certain ingredients of the dentifrice components which enter the gas phase and permeate through the plastic materials of non-gas barrier structures.

The multicompartment container of the present invention has collapsible outer sidewalls and a moveable partition dividing the container into a plurality of compartments whereby the outer sidewalls of the compartments are collapsed to eject, under a compressive force applied to the outer walls, the dentifrice components from the individual compartments with the moveable partition compensating for the deformation of the outer walls of the tube whereby the compressive forces applied to the individual dentifrice components for extrusion thereof from the container are equalized.

IN THE DRAWING

Referring to the accompanying drawing in which corresponding and like parts are designated throughout the several views by the same reference characters.

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Fig. 1 is a side elevation of one embodiment of the multicompartment container, a collapsible tube, used in the practice of the present invention.

Figure 2 is a vertical longitudinal sectional view taken along line 2-2 of Figure 1 of the multicompartmented tube.

Figure 3 is a transverse sectional view, along line 2-2 of Figure 2 with the tube in uncollapsed condition.

Figure 4 is a view of the tube section shown in Fig. 3 in a collapsed condition.

In Figure 1 there is illustrated a multicompartmented tube useful for storing and physically segregating dentifrice components which contain reactive ingredients. The tube shown in Figs. 2-4 includes a tubular body 10 which is shown with a circular section but it could have another shape for example, oval. The tube has a body 10 which is of a type that is easily squeezable, compressible or collapsible and is comprised of a sidewall 12 formed from a resilient plastic material that can be deflected by Deflective Force of about 0.45 to about 1.35 kg as hereinbefore described. One end 20 of the body 10 is sealed while the opposing end 22 is open and is provided with a neck 22a through which the components of the multicomponent dentifrice are discharged through apertures 23 and 23a. The neck 22a is provided with engaging means to accept a closure cap (not shown).

The thickness of sidewall 12 will typically be in the range of about 0.25 to about 1.20 mm. In a preferred embodiment of the invention, the thickness of the sidewall 12 is about 0.25 to about 0.35 mm in thickness.

In the embodiment illustrated, the interior of the body 10 is segmented longitudinally into two compartments 14 and 16 by means of a membrane or partition 18. The partition 18 is flaccid and laterally moveable. Depending upon the construction of the body 10, the partition 18 may be integrally formed with walls of the body 10 as a uniform part of the body 10. Alternatively, the partition 18 may be a separately formed member appropriately retained within the container body. In particular, when the

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body 10 and partition 18 are fabricated from a thermoplastic material such as polyethylene, heat or adhesive sealing may be used to join the two halves of the body 10 together with the partition 18 joined to the peripheral margins of the sidewall 12. The peripheral edges of the partition 18 are held permanently maintained to the internal surface sidewall 12 by a suitable thermal fusion bond or adhesive bond.

In Figure 2, the partition 18 is shown longitudinally in a corrugated or pleated configuration so that as the outer walls of the tube are compressed or deformed, the partition 18 will be caused to move laterally.

In Fig. 3, the partition 18 is shown in its original, resting position prior to the compression of the container sidewalls 12 and Fig. 4 shows partition 18 displaced laterally to a position shown in phantom lines when the collapsible container sidewalls 12 are compressed to codispense the individual dentifrice components from compartments 14 and 16.

The partition 18 is comprised of a very flexible and flaccid thermoplastic film material such as low density polyethylene or polypropylene or may be a laminate in which a gas barrier layer is sandwiched between opposed polyethylene or polypropylene layer. The partition 18 has a thickness substantially less than that of the plastic material from which the sidewalls 12 are formed and generally has a thickness of from 0.005 to 0.20 mm and preferably about 0.075 to about 0.15 mm.

The bottom 20 of the body 10 may have any suitable configuration. The bottom of the body 10 is sealed by crimping or squeezing together with the walls of the body 10 being adhesively or thermally bonded together and to the internal partition 18.

The compartments 14 and 16 of the container body 10 are filled with the dentifrice components in the usual manner before the end 20 of the body 10 is sealed i.e., the container is provided with a sealing cap (not shown); then inverted and filled through the bottom end. The dentifrice components containing different reactive ingredients will exhibit different flow

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characteristics when a compressive force is applied to the sidewall 12, as the dentifrice components as they flow over the interior sidewall surfaces encounter different, i.e., greater or lesser resistance to flow when compared to the other. To compensate for this difference in flow in response to the compressive force applied, the dentifrice components are formulated to account for this difference so that the compressive force required to cause both components to flow is substantially equivalent, the Compressive Force required typically being in the range of about 0.23 to about 9 kg and preferably about 0.45 to about 4.5 kg. When a compressive force in this range is applied to the container sidewalls **10**, as by squeezing the container sidewalls, the individual dentifrice components are caused to simultaneously flow. The partition 18 will move laterally from its fixed position in response to any pressure differential existing in the tube interior whereby the differences in compressive force are equalized. In the event that the difference in Compressive Force required to extrude the individual components is in excess of 20% and cannot be compensated for dentifrice formulation modification, the difference in compressive force required for extrusion may be adjusted by modification of the geometry of the aperture 23, 23a through which the individual components are extruded.

After the multicompartmented container has been filled, partition 18 is, as shown in Figs. 2 and 3, substantially collapsed so that it can be displaced in either direction by a pressure differential across it. In Fig. 4, the partition 18 is shown as shifting laterally to

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the right in response to a compressive displacement of the left side of the tube which is greater than the compressive displacement of the opposite sidewall.

As the components are extruded out of neck 22a through apertures 23, 23a from compartments 14 and 16, the individual components converge to form a single, banded stream of material. The single stream is convenient and easy to direct with accuracy upon a limited surface area, which in the case of toothpaste components can be neatly and easily applied onto the narrow width of a toothbrush in proportions predetermined to provide optimum therapeutic delivery of the reacted ingredients when the component layers are mixed by brushing the teeth or other dental tissue in the oral cavity.

In operation, container 10 may be squeezed intermittently to dispense small portions of the components of a two component dentifrice housed in compartments 14 and 16. When such containers are grasped by a variety of users having individual grips, such squeezing can develop greater pressure on one component than the other. When this occurs, the pressure differential across partition 18 displaces partition 18 until the pressures equalize. Thus, partition 18 is a pressure responsive displaceable wall for equalizing component pressures whereby a relatively constant proportion of a co-dispensed product components is achieved, as both dentifrice components are formulated to be extrudable by the same compressive force, their relative extrusion rates will be equalized by the moveable partition 18.

The diameter of the emerging single stream may be regulated according to packaging specifications by adjusting the diameter of apertures 23 and 23a . Generally the stream is in the form of a cylindrical ribbon having a diameter of about 0.3 to about 2.0 cm. If different colorants are included in the individual dentifrice components, the multilayer dentifrice component stream emerges from the neck outlet passage as an attractive striped toothpaste.

The following example illustrates but does not limit the invention. Unless otherwise indicated, all percentages are by weight and all temperatures are in °C.

Example

A series of 40 mm diameter dual compartmented tubes having a body length of 158 mm was formed from a laminated sheet having the structure PE/EVOH/PE wherein PE is polyethylene and EVOH is an ethylene/vinyl alcohol copolymer, the sheet having a

thickness of 0.3 mm. When tested, a Deflective Force of 0.72 kg was required to deflect the sheet as determined using the Instron® Machine in accordance with the procedure previously described.

The tube was segmented longitudinally into two compartments of approximately similar volume by means of a pleated partition film formed from polyethylene, having a thickness of 0.12 mm which deflected upon the application of a Deflective Force of 0.0347 kg.

The compartments of the dual compartmented tubes were filled with dentifrice components designated 1 and 2 of a multicomponent dentifrice designated "Dentifrice A" which contained the ingredients listed in Table I.

The viscosities of the two dentifrice components when measured in Brookfield Units using a Brookfield® RVT Viscometer-E spindle at 5 rpms were substantially equivalent, the viscosity of component 2 being 17% less than component 1.

TABLE I

Dentifrice A		
Ingredient	Component 1 Wt. % Silica Formula	Component 2 Wt. % Dical Formula
Sorbitol (70%)	36.36	--
Glycerine	10.00	22.0
Polyethylene glycol-600	3.00	--
Carrageenan gum	0.60	0.92
Avicel RC591-F	0.30	--
Tetrasodium pyrophosphate	0.50	0.25
TiO_2	0.30	--
Sodium saccharin	0.25	0.20
NaF	0.510	--

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Dicalcium Phosphate	--	48.76
Silica abrasive	22.0	--
Silica thickener	2.50	--
Color	0.03	--
Flavor oil	0.95	0.89

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Sodium lauryl sulfate	1.20	1.20
Deionized water	Q.S.	Q.S.
Viscosity, Brookfield Units	42	36

The Compressive Force, as determined by the procedure previously described, to cause components 1 and 2 of Dentifrice A was substantially equivalent, that is, the Compressive Force required to extrude component 1 of Dentifrice A was 2.4% less than component 2. The Compressive Forces required to extrude components 1 and 2 of Dentifrice A are recorded in Table III below.

For purposes of comparison the procedure of the Example was repeated except a comparative multicomponent dentifrice designated "Component B" was used. The ingredients of components 1 and 2 of comparative Dentifrice B and their viscosities are recorded in Table II below.

The Compressive Force required for extrusion of the individual components of Dentifrice B were not within ±20% of each other, namely component 2 of Dentifrice B required a Compressive Force which was 45% lower than that of component 1. The Compressive Force required for extrusion of the components of Dentifrice B are recorded in Table III below.

The viscosity of component 2 of comparative Dentifrice B was substantially equivalent to that of component 1, component 2 having a viscosity that was 12% lower than that of component 1.

TABLE II

Dentifrice B		
Ingredient	Component 1 Wt. %	Component 2 Wt. %
NaF	--	0.486
Saccharin	0.20	0.20
Sorbitol	--	38.524
Glycerin	22.00	25.00
Polyethylene glycol 600	--	3.00
Dicalcium phosphate	48.76	--
Silica Abrasive	--	18.0

Silica Thickener	--	5.50
Flavor	0.89	0.89
Sodium lauryl sulfate	1.20	1.20
TiO_2	--	--
Carboxymethyl cellulose	1.00	--
Water	25.75	--
Tetrasodium pyrophosphate	0.25	--
Viscosity, Brookfield Units	65	58

TABLE III

Compressive Force Required for Extrusion (kg)		
	Dentifrice A	Dentifrice B
Component 1	1.14	2.48
Component 2	1.17	4.5

Components 1 and 2 of Dentifrice A were determined to dispense substantially equally form the multicompartment tubes in which they were housed whereas components 1 and 2 Dentifrice B did not, although in both Dentifrices the viscosities of the individual components were substantially equivalent.

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THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A method for the simultaneous coextrusion in controlled equal proportions of reactive ingredients contained in at least two dentifrice components stored and physically segregated in a multicompartmented dispenser body having collapsible body sidewalls, an outlet, and partition means internally dividing the interior volume of the body into a plurality of separated storage compartments, the outlet provided with a discharge aperture in communication with the separate compartments, the partition being formed of a plastic material having a thickness less than that of the body sidewalls and being moveable to compensate for differences in extrusion flow rates of the components upon compression of the sidewalls, the method comprising filling the separate compartments with the individual dentifrice components, the components being formulated to be extrudable form the aperture at substantially equivalent Compressive Forces wherein Compressive Force is measured as the force in kilograms required to extrude 1 to 2 grams of a dentifrice component housed in a stainless steel container a distance of 5 millimeters through an aperture opening of 3.8 mm diameter at a temperature of 23°C at a fixed rate of 150 millimeters/minute (mm/min), the body sidewalls being formed of a plastic material which deforms upon the application of a Deflective Force of about 0.45 or more kilograms, Deflective Force being the maximum force, expressed in kilograms, required to deflect a plastic web bent in the form of an inverted U by a shaped adapter fitted to a compression table, the force being applied axially downward on the arcuate section of the U-shaped web at a rate of 30 cm per minute, and then compressing the sidewalls of the body to extrude from the aperture a single banded miltilayer stream of the components containing the reactive ingredients at ratios predetermined to provide optimum levels of the reacted ingredients when the extruded components are mixed in the oral cavity.

2. The method of claim 1 wherein the Compressive Force causing initiation of the dentifrice component extrusion is within $\pm$ 20% of each other.

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3. The method of claim 1 or claim 2 wherein the dentifrice components are formulated to be extrudable at a Compressive Force of about 0.23 to about 9 kgs.

4. The method of claim 1 or claim 2, wherein the dentifrice components are formulated to be extrudable at a Compressive Force of about 0.45 to about 4.5 kg.

5. The method of claim 1 or claim 2, wherein the plastic material from which the body sidewalls are formed is deformed by a Deflective Force of about 0.45 to about 1.35 kg.

6. The method according to any one of claims 1 to 5, wherein the sidewalls have a thickness of about 0.25 to about 1.2 mm.

7. The method according to any one of claims 1 to 6, wherein the partition is deflected by a Deflective Force of less than 0.23 kgs.

8. The method according to any one of claims 1 to 7, wherein the partition has a wall thickness of about 0.005 to about 0.20 mm.

9. The method according to any one of claims 1 to 8, wherein the partition is pleated and is moveable in response to a compressive force applied to the body sidewalls.

10. The method according to any one of claims 1 to 9, wherein a dentifrice component contains a thickener which imparts elastic structure to the component.

11. The method of claim 10 wherein the thickener is a cellulose gel.

12. The method of claim 10 wherein the thickener is a carrageenan gum.

13. The method according to any one of claims 1 to 12, wherein the reactive ingredients contained in the dentifrice components are calcium containing abrasive and a fluoride releasable salt.

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14. The method of claim 13 or claim 14 wherein the fluoride releasable salt is NaF.

15. The method of claim 13 wherein the calcium abrasive is a calcium phosphate.

16. The method according to any one of claims 1 to 12, wherein the reactive ingredients contained in the dentifrice components are an alkali metal bicarbonate polishing agent and an organic acid.

17. The method of claim 16 wherein the alkali metal bicarbonate is sodium bicarbonate.

18. The method of claim 16 or claim 17 wherein the organic acid is citric acid.

19. The method according to any one of claims 1 to 12, wherein the reactive ingredients contained in the dentifrice components are an alkali metal bicarbonate and a peroxide compound.

20. The method of claim 19 wherein the peroxide compound is hydrogen peroxide or calcium peroxide.

21. The method according to any one of claims 1 to 12, wherein the reactive ingredients contained in the dentifrice components are a calcium containing abrasive and potassium nitrate.

22. The method according to any one of claims 1 to 12, wherein the reactive ingredients contained in the dentifrice components are a calcium containing abrasive and a polycarboxylated polymer.

23. The method according to any one of claims 1 to 12 wherein the reactive ingredients contained in the dentifrice components are a peroxide compound and sodium tripolyphosphate.

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24. The method according to any one of claims 1 to 12 wherein the reactive ingredients contained in the dentifrice components are a peroxide compound and a flavor compound.

25. The method according to any one of claims 1 to 12 wherein the reactive ingredients contained in the dentifrice components are a peroxide compound and a vitamin.

26. The method according to any one of claims 1 to 12 wherein the reactive ingredients contained in the dentifrice components are a peroxide compound and an antibacterial agent.

27. The method according to any one of claims 1 to 12 wherein the reactive ingredients contained in the dentifrice components are a cationic compound and an anionic compound.

Dated this twenty ninth day May 2000

Colgate-Palmolive Company
Patent Attorneys for the Applicant:

F B RICE & CO

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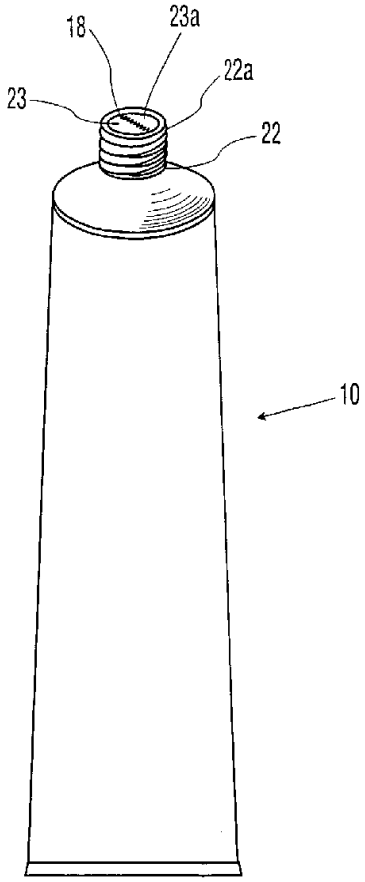

FIG. 1

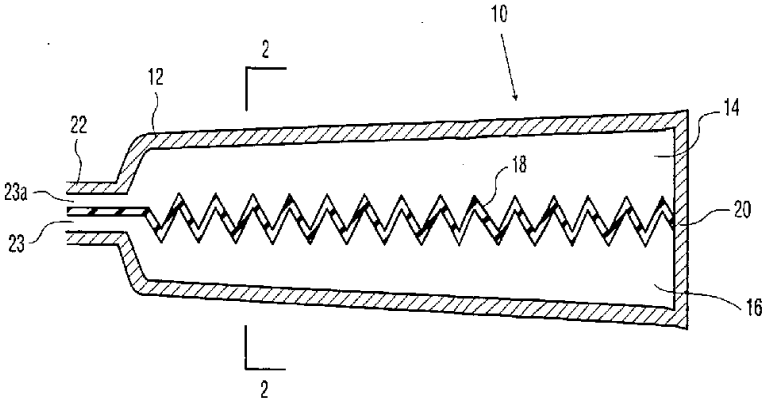

FIG. 2

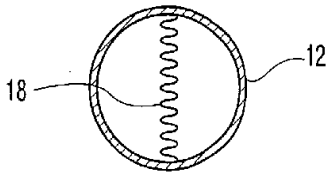

FIG. 3

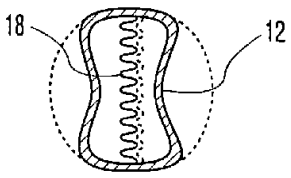

FIG. 4