# **PCT**

# WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



#### INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>6</sup> :		(11) International Publication Number: WO 98/51580
B65D 35/22	<b>A</b> 1	(43) International Publication Date: 19 November 1998 (19.11.98)
<ul> <li>(21) International Application Number: PCT/USS</li> <li>(22) International Filing Date: 30 April 1998 (2)</li> <li>(30) Priority Data: 08/856,065 14 May 1997 (14.05.97)</li> <li>(71) Applicant: COLGATE-PALMOLIVE COMPANY 300 Park Avenue, New York, NY 10022 (US).</li> <li>(72) Inventors: MASTERS, James, G.; 9 Londonder Flemington, NJ 08822 (US). CANADY, Van, Cherry Hill Road, Princeton, NJ 08540 (US). SUI Richard, J.; 1015 Blueberry Court, Edison, NJ 088 (74) Agent: SHAPIRO, Paul; Colgate-Palmolive Comparison River Road, Piscataway, NJ 08855-1343 (US).</li> </ul>	30.04.9 [US/US y Driv L.; 108 LLIVA 317 (US any, 90	BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  Published  With international search report.
(54) Title: CONTAINER HAVING LOW FRICTION IN	IEKNA	AL SIDEWALLS

#### (57) Abstract

A method and container for the simultaneous extrusion in controlled amounts of at least two components of different composition which are stored and physically segregated in a multicompartmented collapsible dispenser, the container sidewalls being formed of a resilient plastic material, the interior component contacting surface of which is formed with a non-migratory material having a low coefficient of friction. When the container sidewalls are compressed there is extruded a single multilayer stream of the components at predetermined volume ratios.

#### FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav	TM	Turkmenistan
BF	Burkina Faso	GR	Greece		Republic of Macedonia	TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel	MR	Mauritania	UG	Uganda
BY	Belarus	IS	Iceland	MW	Malawi	US	United States of America
CA	Canada	IT	Italy	MX	Mexico	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
CG	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	zw	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's	NZ	New Zealand		
CM	Cameroon		Republic of Korea	PL	Poland		
CN	China	KR	Republic of Korea	PT	Portugal		
CU	Cuba	KZ	Kazakstan	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
EE	Estonia	LR	Liberia	$\mathbf{SG}$	Singapore		

#### **CONTAINER HAVING LOW FRICTION INTERNAL SIDEWALLS**

5

#### **BACKGROUND OF THE INVENTION**

#### 1. Field of the Invention

10

The present invention is directed to a multicompartmented container for storing and simultaneously dispensing physically separated components containing ingredients of different adhesive character with respect to the interior sidewalls of the container, at constant predetermined ingredient ratios wherein the interior sidewalls of the container are formed with a non-migratory polymeric material having a low coefficient of friction.

15

#### 2. Prior Art

20

Multicomponent 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, the components being dispensed at specific predetermined ingredient ratios for optimum efficacy and performance.

25

In the dentifrice art, for example, one effective way of depositing anticaries fluoride agents 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<sub>2</sub> 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<sub>2</sub>

30

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<sub>2</sub> and a second component containing a hydrolyzable complex fluoride compound such as sodium fluorosilicate (Na<sub>2</sub>SiF<sub>6</sub>) 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 Cacomplexing 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<sub>2</sub>) removes free Ca<sup>2+</sup> from the solution releasing additional free Ca<sup>2+</sup> from the calcium complexing agent, which, in turn, causes additional CaF<sub>2</sub> to precipitate.

The most convenient and effective way cost to dispense physically separated components of multi-component dentifrices is from a collapsible, compartmented plastic tube. Dual compartmented collapsible tubes for the simultaneous extrusion of two 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 single or multilayer plastic sheet formed from such synthetic polymeric materials as one or more polyethylenes or polypropylenes with or without barrier materials. 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.

A disadvantage experienced by the prior art with two component dispensing systems is disclosed in US 4,481,757 and 4,687,663 namely, that efforts to utilize such systems with semi-solid, extrudable oral care products such as toothpastes and gels containing reactive ingredients which require constant predetermined ingredient ratios to achieve optimum performance, result in unequal dispensing of the dentifrice components from the compartmented tubes 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 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.

5

10

15

20

25

30

35

Factors presently identified as being responsible for the unequal dispensing of the components of multilayer dentifrices for example, in which each component contains a different ingredient such as an abrasive material such as silica or calcium phosphate are the textural attributes that result from the presence of these abrasives in the dentifrice components. In addition, dentifrice components containing the different ingredients interact differently with the polymeric composition of the walls of the dual compartment container, most notably that dentifrice components containing silica abrasives exhibit a greater adhesive character and higher coefficients of static friction with respect to polymeric surfaces such as polyethylene or polypropylene than do dentifrices prepared using dicalcium phosphate abrasives. When a collapsible tube formed of a polymeric material such as polyethylene is used to package the dentifrice components, the adhesive characteristic affects the extrusion force required to extrude the dentifrice from the container as well as the suckback rate after the applied compressive stress is removed resulting in the dispensing of unequal proportions of the desired components.

Therefore, there is a need in the art for a collapsible multi- compartmented container or tube for storing and simultaneously dispensing physically separated dentifrice components containing different ingredients such as abrasives at constant predetermined ingredient ratios whereby optimum proportions of the ingredients are obtained upon mixing the components after extrusion thereof from the container.

#### **SUMMARY OF THE INVENTION**

In accordance with the present invention there is provided a means for the consistent codispensing from a collapsible dual compartmented container or tube, in predetermined proportions of physically separated product components containing ingredients having different adhesive characteristics with respect to the interior sidewalls of a multicompartmented container, the container including a collapsible body portion provided with a partition within the container body defining separate compartments, the

contact surfaces of the inner sidewalls of the container being formed with a non-migratory layer of a low coefficient of friction, whereby substantially constant codispensing of the dentifrice components in predetermined proportions is attained.

5

When the compartments of the multicompartmented container are filled with dentifrice products wherein the inner container sidewalls are formed having a non-migratory slip imparting layer such as Teflon, the surface characteristics of the sidewalls are altered whereby surface interaction between the sidewall contact surfaces and the dentifrice ingredients is equilibrated so that dentifrice components containing different ingredients exhibit similar resistance to slide across the sidewall surfaces thereby providing more even dentifrice component dispensing rates.

### **DESCRIPTION OF THE PREFERRED EMBODIMENTS**

15

10

The term "non-migratory surface" as used herein means a material surface which is non-mobile with respect to the bulk matrix to which it is attached or adhered, wherein the surfaces do not migrate from their substrate and do not become a physical component of the products which contact their exposed surface.

20

25

Materials which form non-migratory surfaces and which exhibit low coefficients of friction include polysiloxanes, graphitic polymers and fluoropolymers. It is preferred that such materials have a coefficient of friction less than about 0.3 as determined by ASTM TEST D1894-78. To maintain the inner wall surfaces of the container at a coefficient of friction of less than about 0.3, the walls are desirably formed with a fluropolymer material such as polytetrafluoroethylene (Teflon) which has a coefficient of friction of less than about 0.2. Polytetrafluoroethylene materials having a coefficient of friction in the range of about 0.04 to about 0.1 are available commercially from E.I. Dupont de Nemours under the trademarks Teflon and Silverstone. Teflon has a coefficient of friction of about 0.04. Silverstone has a coefficient of friction of about 0.1.

30

Other fluoropolymers useful in the practice of the present invention include fluoropolymers of the type disclosed in US 4,595,613 wherein there is disclosed coating the innermost layer of a collapsible dispensing container of laminated wall construction with a fluoroplymer having low flavor absorption properties such as copolymers of hexafluoropropene and tetrafluoroethylene, copolymers of heptafluoropropyl trifluorovinyl ether and tetrafluoroethylene, polychlorotrifluoroethylene and polyvinylidene fluoride.

Additionally, partial or total gaseous fluorination of polyolefin surfaces will also form such fluoropolymer surfaces.

A convenient method of forming the inner contact sidewalls of the compartments of the dual compartmented container with a fluoropolymer is by applying thereto an aqueous or nonaqueous solution coating formulation the sidewalls with fluoropolymers ranging in molecular weight from 2000 to over 1,000,000/monomer repeat units yielding a surface coating of varying thickness and generally about 0.005 to about 3.0 microns in thickness.

10

15

20

25

5

The material having a low coefficient of friction can also be physically blended as a minor, incompatible component of a larger bulk matrix whereby the minor component will migrate to the surface from within the bulk matrix. Examples of such materials include various waxes, and low molecular weight silicone polymers and fluoropolymers and mixtures thereof with polyolefin resins such as ethylene and propylene homopolymers and copolymers. Other means include physical blending of partially compatible materials. In these blends, the surface migration of the minor component is temperature and/or time dependent such as slip additives added to polyolefin resins or the physical blending of compatible materials having significantly different viscosities, examples which include a plasticized polyvinyl chloride resin in which the plasticizer is compatible with the resin, but due to its lower viscosity or molecular weight, migrates to the resin surface over a period of time or the physical blending of high (HMW) or ultra high molecular weight (UHMW) polar polymers with non-polar polymers, examples of which include the Dow-Corning commercially available blends of UHMW polysiloxanes with polyethylene, polypropylene and styrenebutadiene rubbers. The HMW or UHMW material's molecular weight and polymer chain entanglement prevents migration in the solid state but permits migration in the melt phase of the blend.

30

35

Materials from which the body sidewalls are manufactured may be made of aluminum or any suitable plastic material such as polyethylene (both low and high density), polypropylene, ethylene and propylene copolymers butene-1 and propylene copolymers, and polyethylene terephthalate. The material may have a laminate structure wherein a gas barrier material such as ethylene vinyl alcohol, nylon aluminum, silica or other ceramic coating 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.

Multicompartmented containers in which the present invention may be practiced include those having 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, individual components from the individual compartments with a 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 further equalized. A multicompartment tube of this type preferred for use in extruding a multilayer dentifrice in an attractive striped dentifrice stream is disclosed in copending patent application US Serial No. 08/659,737 filed June 6, 1996.

In the preparation of dentifrice components to be used with the multicompartmented containers of the present invention, the dentifrice vehicle used to prepare the dentifrice components is adjusted to impart to the dentifrice component the pasty consistency, body and non-tacky nature which is characteristic of conventional toothpastes and gels. The vehicle is non-reactive with the reactive ingredients of the dentifrice and includes water, a suitable humectant such as glycerin, sorbitol, 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 examples of which include iota carrageenan, kappa carrageenan, lamda carrageenan and mixtures thereof, xanthan gum, carboxymethyl cellulose, sodium carboxymethyl cellulose, starch, 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 trademark Avicel which generally contain about 80 to about 90% by weight microcrystalline cellulose and about 10 to about 20% by weight cellulose gum. A cellulose gel particularly preferred for use in the practice of the present invention is Avicel RC-591-F which is a spray dried cellulose. 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 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 dispensed with the containers 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.

Examples of suitable the surfactants include salts of the higher alkyl sulfates, such as sodium lauryl sulfate or other suitable alkyl sulfates 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 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.

Milticompartmented containers of the present invention are particularly useful for dispensing 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) and calcium and silica abrasives.

5

10

15

20

25

30

35

Abrasive ingredients such as dicalcium phosphate, silica, calcined alumina and alkali metal bicarbonate salt such as sodium bicarbonate are incorporated in the dentifrice components at a concentration of about 20% by weight to about 60% by weight and preferably about 25 to about 55% by weight.

A striped dentifrice product is obtained using the multicompartmented containers of the present invention wherein colorants of contrasting colors are incorporated in each of the dentifrice components to be dispensed, 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 concentration of the dye in the dentifrice composition in an amount from about 0.0005 percent to about 2 percent by weight.

In the dispensing of striped dentifrices, the colorant desirably included in one of the dentifrice components is pigment such as TiO<sub>2</sub> and the colorant distributed throughout the body of the other dentifrice component is a dye of a different color than the pigment included in the first dentifrice component.

A suitable flavoring or sweetening material may also be included in a dentifrice component. 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 dentifrice components.

Various other materials may be incorporated into the dentifrice components including 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.

15

10

5

To prepare the individual dentifrice components, 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 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.

#### **Example**

25

20

Toothpaste tubes of the type described in copending patent application Serial No. 08/659,734, were formed from a laminated sheet having the structure PE/EVOH/PE wherein PE is polyethylene and EVOH is an ethylene/vinyl alcohol copolymer, the tube being segmented longitudinally into two compartments of the same volume by means of a pleated partition film formed from polyethylene.

30

The internal sidewalls of the tubes were in accordance with the practice of the present invention, solution coated a layer of Teflon by pouring a solution containing Teflon into the tube interior, and after a short contact time with the interior sidewalls of the tubes, the Teflon solution was removed, and the interior walls of the tube then allowed to stand at room temperature to allow the solvent to evaporate whereby a dry coating forms on the interior surfaces of the container sidewalls. With this coating process, the coating thickness of the Teflon varied from 0.05 microns to 3.00 microns. The individual

compartments of the internally coated tubes were filled with dentifrice components designated A or B which contained either a silica or dicalcium phosphate abrasive, the amount of the dentifrice ingredients being listed in Table I below.

5 <u>TABLE I</u>

10

15

	<del></del>	
Ingredient	Dentifrice A	Dentifrice B
	Wt. %	Wt. %
	Silica Formula	Dical Formula
Sorbitol (70%)	36.36	
Glycerine	10.00	22.0
Polyethylene glycol-600	3.00	<del></del>
Carrageenan gum	0.82	0.92
Avicel RC591-F	0.30	
Tetrasodium pyrophosphate	0.50	0.25
TiO <sub>2</sub>	0.30	
Sodium saccharin	0.25	0.20
NaF	0.510	
Dicalcium Phosphate		48.76
Silica abrasive	22.0	
Silica thickener	2.00	
Color	0.03	
Flavor oil	0.95	0.89
Sodium lauryl sulfate	1.20	1.20
Deionized water	Q.S.	Q.S.

The compressive force measured in pounds (lbs.) required to extrude a dual component toothpaste portion from the dual compartmented tubes was measured using an Instron® compression tester wherein the dentifrice filled tubes were placed on a molded plastic holder and the holder placed on the bottom jaw of the Instron® compression tester so that the tube rested in the holder at an angle slightly greater than 100° relative to the upper jaw of the tester. The Instron® tester measured the compressive force required to extrude dentifrice from both tube compartments simultaneously at a compression rate of 150 mm/min. over a distance of 12 to 15 mm. The compressive force exerted at the time at which extrusion was initiated from the tube to maintain product flow was recorded as extrusion force.

For purposes of comparison, the procedure of the Example was repeated except the internal sidewall surfaces of the tube compartments were not coated. The extrusion force required to initiate and maintain extrusion from the comparative uncoated dual compartmented tubes by the Instron® tester is also recorded in Table II below.

5

#### TABLE II

Dentifrice Composition	Extrusion force			
	Teflon Coated Tube	Non-Coated Tube		
A	4.2 lbs.	9.0 lbs.		
В	3.9 lbs.	6.9 lbs.		

10

15

The results recorded in Table II indicate that the extrusion force required to initiate extrusion of Dentifrice compositions A and B from the Teflon coated tubes were quite similar, i.e., within less than 10% of each other, whereas the force required for extrusion from the uncoated tubes containing either Dentifrice A and B were substantially different, that is, about 30% more force was required to extrude Dentifrice A from an uncoated tube than Dentifrice B.

#### Example II

20

The procedure of Example I was repeated using two dual compartmented tubes which were filled with Dentifrices A and B in separate compartments of each of the tubes. Thereafter the tubes were hand squeezed to extrude Dentifrice A and Dentifrice B from their respective compartments to form a 3.5 centimeter length of striped toothpaste ribbon. This length of ribbon was then weighed and the Dentifrice B component was separated from the ribbon and also weighed. By subtracting the weight of Dentifrice B from the weight of the striped ribbon, the weight of Dentifrice A could be calculated. By taking the densities of Dentifrice components A and B into account (e.g., the density for Dentifrice component A was 1.32 grams/milliliter and 1.48 grams/milliliter for component B), weight % was converted to volume %. From this data, the volume % of Dentifrices A and B in the extruded ribbon, could then be determined, and the ratio of these percentages then provided an indication of the preciseness of the dual component extrusion, the results of which are recorded in Table III below.

30

For purposes of comparison, the procedure of Example II was repeated except dual compartmented tubes which had not been Teflon coated were filled with Dentifrices A and B. The volume ratio of the extruded components of this comparative dentifrice are also recorded in Table III below.

5

#### TABLE III

	Weight of I	Dispensed	Wt. of Di	spensed	Compor	nent A/B
Interior Body	Ribbon (	grams)	Component	B (grams)	Volume 1	Ratio (%)
Interior Body Sidewall Condition	Tube #1	Tube #2	Tube #1	Tube #2	Tube #1	Tube #2
Coated	1.7135	2.012	0.9827	1.043	46/54	51/49
Uncoated	1.624	2.000	1.087	1.349	36/64	35/65

10

15

The data in Table III indicate that substantially equal volumes e.g., 46:54 and 51:49 of the Dentifrice components A and B were dispensed from the Teflon coated multicompartment tubes whereas Dentifrice A and B were extruded in widely varying volume proportions from the uncoated tubes.

#### **CLAIMS**

#### What is claimed is:

1. A method for the simultaneous extrusion of a plurality of components of different composition in substantially equal proportions from a collapsible multicompartmented container which method comprises forming the sidewalls of the interior component contacting surfaces of the container sidewalls with a non-migratory material having a low coefficient of friction, filling the individual compartments of the container with the individual components, applying a compressive force to the body sidewalls to extrude single banded multilayer stream of the components whereby the volume ratios of extruded components are at substantially predetermined proportions.

15

5

10

2. The method of claim 1 wherein the interior body sidewalls are formed with a fluoropolymer.

3. The method of claim 2 wherein fluoropolymer has a coefficient of friction less than 0.3.

0.5.

4. The method of claim 2 wherein the fluoropolymer is Teflon.

- 5. The method of claim 1 wherein the container sidewalls are formed from polyethylene and a fluoropolymer.
  - 6. The method of claim 1 wherein the components are dentifrice compositions containing different abrasives.

30

35

25

7. A collapsible dual compartmented container for the simultaneous coextrusion in controlled equal proportions of at least two components the container being comprised of a body having sidewalls and a partition means internally dividing the interior volume of the body into a plurality of separated storage compartments, the body sidewalls being formed of a material which deforms upon the application of a compressive force, the interior component contacting surfaces of the sidewalls being formed with a non-migratory material having a low coefficient of friction so that when a compressive force is applied to

the body sidewalls, the components are extruded at substantially predetermined volume proportions.

- 8. The container of claim 7 wherein the interior body sidewalls are coated with a fluoropolymer.
  - 9. The container of claim 8 wherein fluoropolymer has a coefficient of friction less than 0.3.
    - 10. The container of claim 7 wherein the fluoropolymer is Teflon.

- 11. The container of claim 7 wherein the container sidewalls are formed from polyethylene and a fluoropolymer.
- 15 12. The container of claim 7 wherein the components are dentifrices containing different abrasives.

ernational Application No PCT/US 98/08572

			PC1/US 98/085/2	
A. CLASSIF	FICATION OF SUBJECT MATTER  865D35/22			
1100	0000037 22			
	International Patent Classification (IPC) or to both national classifi	cation and IPC		
B. FIELDS	SEARCHED cumentation searched (classification system followed by classifica	tion oumbolo)		
IPC 6	B65D	tion symbols)		
Documentati	ion searched other than minimum documentation to the extent that	such documents are includ	ed in the fields searched	
Electronic da	ata base consulted during the international search (name of data b	pase and, where practical, s	earch terms used)	
C. DOCUME Category	ENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document, with indication, where appropriate, of the re	elevant passages	Relevant to claim No.	
A	US 4 487 757 A (KIOZPEOPLOU) 11 1984	December	1,7	
	cited in the application			
	see column 3, line 55 - column 4	4, line 51;		
	figures 1-3			
Α	US 4 687 663 A (SCHAEFFER) 18 A	ugust 1987	1,7	
	cited in the application	•	,	
	see column 5, line 23 - line 65	; figures		
Α	US 5 137 178 A (STOKES) 11 Augus	st 1992	1,7	
	cited in the application see column 1, line 29 - line 39	. olaim 1		
		; Claim 1		
		-/		
			ľ	
V Furt	her documents are listed in the continuation of box C.	V Patent family m	embers are listed in annex.	
		X Patent family m	embers are instead in annex.	
· ·	ategories of cited documents :		shed after the international filing date not in conflict with the application but	
consid	ent defining the general state of the art which is not dered to be of particular relevance		I the principle or theory underlying the	
"E" earlier o	document but published on or after the international date	"X" document of particu	lar relevance; the claimed invention ed novel or cannot be considered to	
	ent which may throw doubts on priority claim(s) or is cited to establish the publication date of another	involve an inventive	e step when the document is taken alone lar relevance; the claimed invention	
	n or other special reason (as specified) ent referring to an oral disclosure, use, exhibition or	cannot be consider	rar relevance, the claimed invention red to involve an inventive step when the ned with one or more other such docu-	
other	means ent published prior to the international filing date but		nation being obvious to a person skilled	
	han the priority date claimed	"&" document member of	of the same patent family	
Date of the	actual completion of theinternational search	Date of mailing of th	ne international search report	
1	0 August 1998	17/08/19	998	
Name and	mailing address of the ISA	Authorized officer		
	European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,			
	Fax: (+31-70) 340-2040, 1x: 31 651 epo ni, Fax: (+31-70) 340-3016	Martens, L		

rnational Application No
PCT/US 98/08572

Citation of document, with indication where appropriate, of the relevant passages	Polovant to glaim No.
onation of document. with indication, where appropriate, or the relevant passages	Relevant to claim No.
US 5 020 694 A (PETTENGILL) 4 June 1991 cited in the application see column 1, line 49 - column 2, line 7 see column 2, line 25 - line 41	1,7
US 4 595 613 A (TAVSS) 17 June 1986 cited in the application see the whole document	1,7
	cited in the application see column 1, line 49 - column 2, line 7 see column 2, line 25 - line 41 US 4 595 613 A (TAVSS) 17 June 1986 cited in the application see the whole document

Information on patent family members

rnational Application No PCT/US 98/08572

	tent document in search report		Publication date		Patent family member(s)	Publication date
US 4487757 A		11-12-1984	AU 550400 B AU 9177482 A BR 8207496 A CA 1200799 A CH 660470 A DE 3247353 A DK 576082 A FR 2518960 A GB 2112642 A,B JP 1726961 C JP 4011524 B JP 58149261 A		20-03-1986 07-07-1983 18-10-1983 18-02-1986 30-04-1987 07-07-1983 29-06-1983 01-07-1983 27-07-1983 19-01-1993 28-02-1992 05-09-1983	
US	4687663	A	18-08-1987	US AU CA DE DE EP US BR JP JP JP IN IN	4528180 A 580076 B 1257545 A 3587725 D 3587725 T 0202359 A 4849213 A 4983379 A 8503355 A 1463004 C 61271214 A 63007522 B 166887 A 166888 A 166889 A	09-07-1985 22-12-1988 18-07-1989 24-02-1994 28-04-1994 26-11-1986 18-07-1989 08-01-1991 09-12-1986 28-10-1988 01-12-1986 17-02-1988 04-08-1990 04-08-1990
US	5137178	A	11-08-1992	CA	2062619 A	18-10-1992
US	5020694	A	04-06-1991	AT AU CA DE DE EP JP	139204 T 640636 B 5130390 A 2011971 A,C 69027353 D 69027353 T 0388185 A 2282083 A 2575914 B	15-06-1996 02-09-1993 20-09-1990 16-09-1990 18-07-1996 05-12-1996 19-09-1990 19-11-1990 29-01-1997

Information on patent family members

rnational Application No PCT/US 98/08572

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5020694 A	1	PH 26659 A US 5038963 A	04-09-1992 13-08-1991
US 4595613 A	17-06-1986	AU 564676 B AU 3726385 A BR 8406757 A CA 1222686 A DE 3445798 A DK 618284 A FR 2557502 A GB 2151985 A,B GB 2184394 A,B JP 60228145 A MX 170971 B SE 463908 B SE 8406433 A	20-08-1987 18-07-1985 22-10-1985 09-06-1987 11-07-1985 01-07-1985 05-07-1985 31-07-1985 24-06-1987 13-11-1985 22-09-1993 11-02-1991 01-07-1985