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A B O G A D O S

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**SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO**
**DIVISIÓN DE NUEVAS CREACIONES**
**E. S. D.**

Ref.: Expediente No. 02035233
Solicitud de Patente a nombre de
**Johnson & Johnson Industria e Comercio Ltda.**

Yo, **JIMENA ESCOBAR URIBE**, identificada como aparece al pie de mi firma, domiciliada en Bogotá, en la Carrera 11 A No. 94-76 Of. 305, obrando como apoderada de la sociedad **Johnson & Johnson Industria e Comercio Ltda.**, domiciliada en São Paulo, Brasil, solicitante de la patente de la referencia, pido a usted que se proceda a examinar si la invención, cuya patente se tramita en el expediente de la referencia, es patentable.

Adjunto al presente memorial el recibo No. 04 - 76,748 de septiembre 28, 2004, por la suma de $353.000.oo, con lo cual queda cubierta la tasa oficial correspondiente a dicho estudio.

Señor Superintendente,

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(54) Title: SYSTEMS COMPRISING ORGANOSILOXANE RESINS FOR DELIVERING ORAL CARE SUBSTANCES AND FOR PROLONGING SUCH DELIVERY

(57) Abstract: Disclosed is a system for delivering an oral care substance to the oral cavity comprising: (a) a delivery composition comprised of: (i) an organosiloxane resin; (ii) a volatile carrier capable of solubilizing the organosiloxane resin; (iii) a rheology modifier; and (iv) at least one oral care substance; and (b) a protective composition comprised of: (i) an organosiloxane resin; and (ii) a volatile carrier capable of solubilizing the organosiloxane resin. Further disclosed is a system for delivering an oral care substance to the oral cavity comprising: (a) a delivery composition comprised of: (i) an organosiloxane resin; (ii) a fluid diorganopolysiloxane-based polymer; (iii) a volatile carrier capable of solubilizing the organosiloxane resin and the fluid diorganopolysiloxane-based polymer; (iv) a rheology modifier; and (v) at least one oral care substance; and (b) a protective composition comprised of: (i) an organosiloxane resin; and (ii) a volatile carrier capable of solubilizing the organosiloxane resin. The protective composition may further comprise a fluid diorganopolysiloxane-based polymer and/or a rheology modifier. Still further disclosed is a method of using these systems.

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# SYSTEMS COMPRISING ORGANOSILOXANE RESINS FOR DELIVERING ORAL CARE SUBSTANCES AND FOR PROLONGING SUCH DELIVERY

## FIELD

The present invention relates to systems for delivering oral care substances to the surfaces of the teeth and for prolonging such delivery. More specifically, the present invention relates to a system comprising a delivery composition comprising organosiloxane resins and at least one oral care substance for delivering the oral care substance, and a protective composition comprised of organosiloxane resins for prolonging the presence of the delivery composition in the oral cavity. The delivery composition forms a film on the surface to which it has been applied and provides sustained release of the oral care substance from the film for prolonged therapeutic, prophylactic, and/or cosmetic benefits. In addition, it is believed that the systems herein may further provide sustained release benefits to other oral surfaces, such as the gingival and mucosal tissues, as well as to the surfaces of the teeth.

## BACKGROUND

Oral care products by which various oral care substances or actives can be delivered to the soft and hard tissues of the oral cavity have previously been known. Examples of such oral care products include, for example, brushing aids such as dentifrice products for delivery of anti-caries actives such as fluoride or other actives for the reduction of the bacteria that lead to the formation of plaque, and mouthwashes containing breath freshening actives and/or anti-bacterial actives. In addition, bleaching agents such as peroxide that can be applied directly to the surfaces of the teeth, i.e., to the tooth enamel, have been developed.

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While the specification concludes with claims particularly pointing out and distinctly claiming the invention, it is believed that the present invention will be better understood from the following description.

All percentages and ratios used hereinafter are by weight of total composition, unless otherwise indicated.

All measurements referred to herein are made at 25°C unless otherwise specified.

All percentages, ratios, and levels of ingredients referred to herein are based on the actual amount of the ingredient, and do not include solvents, fillers, or other materials with which the ingredient may be combined as a commercially available product, unless otherwise indicated.

All publications, patent applications, and issued patents mentioned herein are hereby incorporated in their entirety by reference. Citation of any reference is not an admission regarding any determination as to its availability as prior art to the claimed invention.

Herein, "comprising" means that other steps and other components which do not affect the end result can be added. This term encompasses the terms "consisting of" and "consisting essentially of."

The delivery systems herein are comprised of a delivery composition and a protective composition. The delivery and protective compositions herein comprise essential components, as well as optional components. The essential and optional components of these compositions of the present invention are described in the following paragraphs.

## DELIVERY COMPOSITION

The delivery composition herein is a composition for delivering an oral care substance to the oral cavity. The delivery composition comprises an oral care substance for providing a therapeutic, prophylactic, and/or cosmetic benefit to the oral cavity. One preferred embodiment of the delivery composition is comprised of an organosiloxane resin; a volatile carrier capable of solubilizing the organosiloxane resin; a rheology modifier; and at least one oral care substance.

Another preferred embodiment of the delivery composition is comprised of an organosiloxane resin; a fluid diorganopolysiloxane-based polymer; a volatile carrier capable of solubilizing the organosiloxane resin and the fluid

diorganopolysiloxane-based polymer; a rheology modifier; and at least one oral care substance.

PROTECTIVE COMPOSITION

The protective composition acts as a protective coating to prolong the presence of the oral care substance or substances contained in the delivery composition in the oral cavity. The protective composition need not comprise an oral care substance.

One preferred embodiment of the protective composition is comprised of an organosiloxane resin and a volatile carrier capable of solubilizing the organosiloxane resin. A rheology modifier may also be present.

Another preferred embodiment of the protective composition is comprised of an organosiloxane resin, a fluid diorganopolysiloxane-based polymer, a volatile carrier capable of solubilizing the organosiloxane resin and the fluid diorganopolysiloxane-based polymer. A rheology modifier may also be present.

The protective composition need not comprise an oral care substance in order to act as a protective coating for the delivery composition. However, it should be noted that any of the oral care substances described herein could additionally be added to the protective composition.

It should also be noted that more than one application of the protective composition, following the application of the delivery composition, can be made. Multiple applications of the protective composition can be made and are within the scope of the present invention.

Organosiloxane Resins

Silicone resins are highly crosslinked polymeric siloxane systems. The crosslinking is introduced through the incorporation of tri-functional and tetra-functional silanes with mono-functional or di-functional, or both, silanes during manufacture of the silicone resin. As is well understood in the art, the degree of crosslinking that is required in order to result in a silicone resin will vary according to the specific silane units incorporated into the silicone resin. In general, silicone materials which have a sufficient level of trifunctional and tetrafunctional siloxane monomer units, and hence, a sufficient level of crosslinking, such that they dry down to a rigid, or hard, film are considered to be silicone resins. The ratio of oxygen atoms to silicon atoms is indicative of the level of crosslinking in a particular silicone material. Silicone materials which

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have at least about 1.1 oxygen atoms per silicon atom will generally be silicone resins herein. Preferably, the ratio of oxygen:silicon atoms is at least about 1.2:1.0.

Silicone materials and silicone resins in particular can conveniently be identified according to a shorthand nomenclature system well known to those skilled in the art as the "MDTQ" nomenclature. Under this system, the silicone is described according to the presence of various siloxane monomer units which make up the silicone. Briefly, the symbol M denotes the mono-functional unit $(CH_3)_3SiO_{.5}$; D denotes the difunctional unit $(CH_3)_2SiO$; T denotes the trifunctional unit $(CH_3)SiO_{1.5}$; and Q denotes the quadra- or tetra-functional unit $SiO_2$. Note that a small amount, up to about 5% of silanol or alkoxy functionality may also be present in the resin structure as a result of processing.

Primes of the unit symbols, e.g., M', D', T', and Q', denote substituents other than methyl, and must be specifically defined for each occurrence. Typical alternate substituents include groups such as vinyl, phenyl, amino, hydroxyl, etc. The molar ratios of the various units, either in terms of subscripts to the symbols indicating the total number of each type of unit in the silicone, or an average thereof, or as specifically indicated ratios in combination with molecular weight, complete the description of the silicone material under the MDTQ system. Higher relative molar amounts of T, Q, T' and/or Q' to D, D', M and/or M' in a silicone resin is indicative of higher levels of crosslinking. As discussed before, however, the overall level of crosslinking can also be indicated by the oxygen to silicon ratio.

The organosiloxane resins are solid at about 25°C and the average molecular weight of the resins is from about 1,000 to about 10,000. The resins are soluble in organic solvents such as toluene, xylene, isoparaffins, and cyclosiloxanes or the volatile carrier described below, indicating that the resin is not sufficiently crosslinked such that the resin is insoluble in the volatile carrier.

The silicone resins preferred for use herein are MQ, MT, MTQ, and MDTQ resins; such MQ resins are disclosed in U.S. Patent 5,330,747, Krzysik, issued July 19, 1994. Thus, the preferred silicone substituent is methyl. Especially preferred are MQ resins wherein the M:Q ratio is from about 0.5:1.0 to about 1.5:1.0. Organosiloxane resins such as these are commercially available, for example, Wacker 803 and 804 available from Wacker Silicones Corporation of Adrian, Michigan, US, and G.E. 1170-002 from the General Electric Company.

Baker of Phillispburg, N.J, and HFE (a mixture of methyl nonafluoroisobutyl ether and methyl nonafluorobutyl ether), supplied by the 3M Company.

Rheology Modifiers

The compositions further comprise a rheology modifier which inhibits settling and separation of components or controls settling in a manner which facilitates re-dispersion and may control rheological flow properties. Suitable rheology modifiers herein include organo modified clays, silicas, polyethylene, and mixtures thereof. The preferred organophilic clays comprise quaternium-18 hectorite or Stearalkonium hectorite, such as Bentone 27 and 38™ from Rheox, organoclay dispersion such as Bentone ISD gel ™; or bentonite organo modified clays such as Bentone 34 ™ from Rheox or the Claytone Series ™ from Southern Clay Products; and mixtures thereof. The preferred silicas may be fumed silica such as the Aerosil ™ series from Degussa or the Cab-o-sil ™ series from Cabot Corporation, silica gels such as the Sylodent ™ or Sylox ™ series from W. R. Grace & Co. or precipitated silica such as Zeothix 265 from J. M. Huber Corporation.

The rheology modifier is preferably present in the composition at a level of from about 0.1% to about 30%, preferably from about 0.5% to about 10%, and even more preferably about 1% to about 3% of the composition.

Oral Care Substances

The oral care substance preferably contains an active at a level where upon directed use, the benefit sought by the wearer is promoted without detriment to the oral surface to which it is applied. Examples of the oral conditions these actives address include, but, are not limited to, appearance and structural changes to teeth, whitening, stain bleaching, stain removal, plaque removal, tartar removal, cavity prevention and treatment, inflamed and/or bleeding gums, mucosal wounds, lesions, ulcers, aphthous ulcers, cold sores, tooth abscesses, and the elimination of mouth malodor resulting from the conditions above and other causes such as microbial proliferation.

Suitable oral care substances include any material that is generally considered safe for use in the oral cavity and that provides changes to the overall appearance and/or health of the oral cavity. The level of oral care substance in the compositions of the present invention is generally, unless specifically noted, from about 0.01% to about 50%, preferably from about 0.1%

fluoride ion-yielding materials are found in Briner et al; U.S. Pat. No. 3,535,421; issued Oct. 20, 1970 and Widder et al; U.S. Pat. No. 3,678,154; issued July 18, 1972. Preferred fluoride ion sources for use herein include sodium fluoride, potassium fluoride and ammonium fluoride. Sodium fluoride is particularly preferred. Preferably the instant compositions provide from about 50 ppm to 10,000 ppm, more preferably from about 100 to 3000 ppm, of fluoride ions in the compositions that contact dental surfaces when used with the delivery system of the present invention.

4. Anti-microbial Agents

Anti-microbial agents can also be present in the oral care compositions or substances of the present invention. Such agents may include, but are not limited to, 5-chloro-2-(2,4-dichlorophenoxy)-phenol, commonly referred to as triclosan, and described in The Merck Index, 11th ed. (1989), pp. 1529 (entry no. 9573) in U.S. Patent No. 3,506,720, and in European Patent Application No. 0,251,591 of Beecham Group, PLC, published January 7, 1988; phthalic acid and its salts including, but not limited to those disclosed in U.S. Pat. 4,994,262, Feb. 19, 1991, preferably magnesium monopotassium phthalate, chlorhexidine (Merck Index, no. 2090), alexidine (Merck Index, no. 222; hexetidine (Merck Index, no. 4624); sanguinarine (Merck Index, no. 8320); benzalkonium chloride (Merck Index, no. 1066); salicylanilide (Merck Index, no. 8299); domiphen bromide (Merck Index, no. 3411); cetylpyridinium chloride (CPC) (Merck Index, no. 2024; tetradecylpyridinium chloride (TPC); N-tetradecyl-4-ethylpyridinium chloride (TDEPC); octenidine; delmopinol, octapinol, and other piperidino derivatives; nicin preparations; zinc/stannous ion agents; antibiotics such as augmentin, amoxicillin, tetracycline, doxycycline, minocycline, and metronidazole; and analogs and salts of the above; essential oils including thymol, geraniol, carvacrol, citral, hinokitiol, eucalyptol, catechol (particularly 4-allyl catechol) and mixtures thereof; methyl salicylate; hydrogen peroxide; metal salts of chlorite and mixtures of all of the above.

5. Anti-inflammatory Agents

Anti-inflammatory agents can also be present in the oral care compositions or substances of the present invention. Such agents may include, but are not limited to, non-steroidal anti-inflammatory agents or NSAIDs such as ketorolac, flurbiprofen, ibuprofen, naproxen, indomethacin, aspirin, ketoprofen, piroxicam and meclofenamic acid. Use of NSAIDs such as Ketorolac are claimed in U.S. Patent 5,626,838, issued May 6, 1997. Disclosed therein are

Anti-pain or desensitizing agents can also be present in the oral care compositions or substances of the present invention. Such agents may include, but are not limited to, strontium chloride, potassium nitrate, natural herbs such as gall nut, Asarum, Cubebin, Galanga, scutellaria, Liangmianzhen, Baizhi, etc.

11. Anti-viral Actives

Antiviral actives useful in the present composition include any know actives that are routinely use to treat viral infections. Such anti-viral actives are disclosed in Drug Facts and Comparisons (loose leaf drug information service), Wolters Kluer Company, St. Louis, Mo., ©1997, pp. 402(a)-407(z), incorporated herein by reference in its entirety. Specific examples include anti-viral actives disclosed in U.S. Patent 5,747,070, issued May 5, 1998 to Satyanarayana Majeti, incorporated herein by reference in its entirety. Said Patent discloses the use of stannous salts to control viruses. Stannous salts and other anti-viral actives are described in detail in Kirk & Othmer, Encyclopedia of Chemical Technology, Third Edition, Volume 23, Wiley-Interscience Publishers (1982), pp. 42-71, incorporated herein by reference in its entirety. The stannous salts that may be used in the present invention would include organic stannous carboxylates and inorganic stannous halides. While stannous fluoride may be used, it is typically used only in combination with another stannous halide or one or more stannous carboxylates or another therapeutic agent.

12. Other Ingredients

In addition to the above materials of the composition of the present invention, a number of other components may desirably be added to the oral care substance. Additional components include, but are not limited to, flavoring agents, sweetening agents, xylitol, opacifiers, coloring agents, surfactants, and chelants such as ethylenediaminetetraacetic acid. Suitable flavoring agents include, but are not limited to, oil of peppermint, oil of sassafras, clove bud oil, peppermint, menthol, anethole, thymol, methyl salicylate, eucalyptol, cassia, 1-menthyl acetate, sage, eugenol, parsley oil, oxanone, oil of wintergreen, alpha-irisone, oil of spearmint, marjoram, lemon, orange, propenyl guaethol, cinnamon, and mixtures thereof.

Pigments may also added to the compositions herein to more precisely indicate the locations at which the composition has actually been applied, allowing the user to apply the composition more thoroughly or evenly. However, such pigments are not intended to mask stains that may exist on the tooth surfaces.

These additional ingredients can also be used in place of the compounds disclosed above.

## EXAMPLES

The following examples further describe and demonstrate embodiments within the scope of the present invention. The examples are given solely for the purpose of illustration and are not to be construed as limitations of the present invention, as many variations thereof are possible without departing from the spirit and scope of the invention.

Table 1: Hydrophobic Delivery Composition

| Component | Ex.1 | Ex.2 | Ex.3 | Ex.4 | Ex.5 | Ex.6 |
|---|---|---|---|---|---|---|
| Organosiloxane Resin[1] | 25 | 25 | 24.6 | 25 | 25 | 25 |
| Silicone Gum[2] | 12.5 | 4.2 | 3.5 | 2.5 | 1.8 | -- |
| Oral Care Substance[3] | 17 | 17 | 17 | 17 | 17 | 17 |
| Volatile Carrier[4] | 44.5 | 52.8 | 53.9 | 54.5 | 55.2 | 57.0 |
| Bentone Clay[5] | 1 | 1 | 1 | 1 | 1 | 1 |
| TOTAL | 100 | 100 | 100 | 100 | 100 | 100 |

1. E.g., MQ resin available as 1170-002 from General Electric.
2. E.g., Dimethicone gum available as SE 30 or SE 63 from General Electric.
3. E.g., Sodium percarbonate as a dry powder.
4. E.g., Isododecane or a 50/50 mixture of ethyl acetate and butanone or a mixture of ethyl acetate, propyl acetate and HFE. The percentage of each individual solvent component in the mixed solvent systems can vary from 0 to 100%, respectively.
5. Bentone 27 available from Rheox.

Table 2: Hydrophobic Delivery Composition

| Component | Ex.1 | Ex.2 | Ex.3 | Ex.4 |
|---|---|---|---|---|
| Organosiloxane Resin[1] | 33.43 | 33.43 | 35.14 | 33.43 |
| Silicone Gum[2] | 5.57 | 5.57 | 5.86 | 5.57 |
| Oral Care Substance[3] | 19.00 | 12.00 | 19.00 | 19.00 |
| Ethyl acetate | 8.00 | 8.50 | 8.00 | 8.00 |

| | | | | |
|---|---|---|---|---|
| Bentone Gel[4] | 10.00 | 10.00 | 10.00 | 10.00 |
| DC-200/350cst.[5] | 1.00 | 1.00 | 2.00 | 1.00 |
| HFE-7100[6] | 21.00 | 26.00 | - | 19.50 |
| N-propyl acetate | 2.00 | 2.00 | - | 2.00 |
| 2-butanone | - | - | 19.00 | - |
| Aerosil 200[7] ($SiO_2$) | - | - | 1.00 | - |
| Flavor | - | 1.50 | - | 1.50 |
| TOTAL | 100 | 100 | 100 | 100 |

1. E.g., MQ resin available as 1170-002 from General Electric.
2. E.g., Dimethicone gum available as SE 30 from General Electric.
3. E.g., Sodium percarbonate as a dry powder.
4. Bentone Gel IPM available from Rheox.
5. DC-200/350 is: dimethylpolysiloxane, CAS# 9016-00-6, from Dow Corning.
6. HFE-7100 is a mixture of Methyl Nonafluoroisobutyl Ether, CAS# 163702-08-7 and Methyl Nonafluorobutyl Ether, CAS# 163702-07-6, manufactured by 3M Co.
7. Aerosil 200 is silicon dioxide (chemically prepared), CAS# 112945-52-5, from Degussa AG.

Method of Preparation

The delivery compositions of Tables 1 and 2 are non-aqueous. The oral care substances are dispersed or dissolved in a solution comprising the organosiloxane resin, the fluid diorganopolysiloxane-based polymer, the volatile carrier, and the rheology modifier.

The compositions of Tables 1 and 2 are suitably prepared as follows. Three hundred (300) grams of organosiloxane resin solution (for example, 43.7% MQ resin solution in isododecane or in a 50/50 mixture of ethyl acetate and butanone (or in a mixture of ethyl acetate, propyl acetate and HFE) are mixed with 147.30 grams of fluid diorganopolysiloxane polymer solution (for example, 50% SE30 silicone gum solution in isododecane or a 50/50 mixture of ethyl acetate and butanone or a mixture of ethyl acetate, propyl acetate and HFE). The oral care substances are then dispersed in the resin/gum mixture. This method may be carried out without the presence of the silicone gum.

All oral care substances described herein can formulated as described above.

Table 5: Oral Care Delivery Compositions

| Component | Ex.1 | Ex.2 | Ex.3 | Ex.4 | Ex.5 | Ex.6 |
|---|---|---|---|---|---|---|
| Ethyl Acetate | 26.00 | 22.00 | 35.00 | 28.00 | 25.77 | 23.00 |
| 2-Butanone | 25.95 | 17.00 | 9.00 | 14.50 | 25.50 | 23.45 |
| Isododecane | – | 10.00 | – | – | 5.00 | – |
| Limonene | – | 4.00 | – | – | – | – |
| MQ Resin | 28.00 | 32.50 | 33.00 | 35.00 | 27.00 | 28.00 |
| SE 30 Silicone Gum | 7.00 | – | 11.00 | 5.00 | 3.00 | 7.00 |
| Silicone Fluid 10cStk | – | 6.50 | – | – | – | – |
| Bentone Gel ISD | 10.00 | – | 8.00 | 10.00 | 10.00 | – |
| Claytone HY | – | 2.00 | – | – | – | 7.00 |
| Cab-o-Sil M7D | – | – | 1.50 | – | – | – |
| Bismuth Oxychloride | – | 5.00 | – | – | – | 10.00 |
| Titanium Dioxide | 3.00 | – | 1.00 | 5.00 | 2.00 | – |
| Flavor Oil | – | – | 0.10 | – | 0.15 | 0.15 |
| Polymethylsilsesquioxane | – | – | 1.00 | – | – | – |
| Polymethylmethyacrylate | 3.00 | 1.00 | – | – | 0.50 | – |
| Nylon 12 | – | – | – | 2.00 | – | 1.00 |
| Silica | – | – | – | – | 0.50 | – |
| FD&C Yellow #5 Al Lake | 0.05 | – | 0.10 | – | 0.05 | 0.10 |
| Iron Oxide, Red | – | – | – | 0.50 | 0.03 | – |
| Sodium Saccharin | – | – | 0.30 | – | 0.50 | 0.30 |
| TOTAL | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |

Table 6: Ethanol Based Delivery Compositions

| Component | Ex.1 | Ex.2 | Ex.3 | Ex.4 | Ex.5 | Ex.6 |
|---|---|---|---|---|---|---|
| Ethanol | 20.90 | 35.80 | 41.70 | 25.89 | 23.10 | 23.55 |
| Ethyl Acetate | – | – | – | – | 4.95 | – |
| Ethyl Butyrate | 10.30 | – | – | 12.74 | 11.75 | 10.9 |
| Isoamyl Acetate | – | – | – | – | – | 2.65 |
| Isododecane | – | 13.00 | – | – | – | – |

| | | | | | | |
|---|---|---|---|---|---|---|
| MQ Resin | 35.10 | 32.85 | 42.75 | 47.00 | 42.00 | 41.80 |
| Silicone Visc-100M | – | – | – | – | 7.15 | – |
| Silicone Fluid 100 cSt | 7.80 | – | – | 9.65 | – | 8.55 |
| Silicone Fluid 10 cStk | 3.90 | – | 11.40 | – | 4.00 | – |
| Silwet L-7500 | – | – | – | – | – | 6.58 |
| Bentone 27 | 1.50 | – | 2.00 | 1.85 | 1.35 | 1.97 |
| Claytone APA | – | 2.00 | – | – | – | – |
| Cab-o-Sil | – | – | – | – | 0.45 | – |
| Sodium Percarbonate | 19.00 | – | – | – | – | – |
| Carbamide Peroxide | – | 15.00 | – | – | – | – |
| Potassium Nitrate | – | – | – | – | 5.00 | – |
| Tripolyphosphate | – | – | – | – | – | 2.50 |
| Chlorhexidine Digluconate | – | – | – | 2.57 | – | – |
| Titanium Dioxide | 1.50 | – | 2.00 | – | – | 1.50 |
| Flavor Oil | – | – | 0.05 | – | – | – |
| Sodium Fluoride | – | – | 0.10 | – | 0.25 | – |
| Sodium Saccharin | – | 0.20 | – | 0.30 | – | – |
| | | | | | | |
| | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |

Method of Preparation

The delivery compositions of Tables 3-6 are suitably prepared as follows. Add the solvents into a container suitable to minimize solvent loss. Add the rheology modifiers and mix until well dispersed. Add the silicone resin and mix until completely dissolved. Add the silicone gum and/or silicone fluids and mix until completely dissolved. At this time add any salts such as sodium percarbonate and/or other oral care actives, aesthetic ingredients such as opacifiers, sweeteners, dyes, and flavors. Continue mixing until homogeneous. Additional high shear mixing may be used to promote the mixing. Pack into airtight containers.

Alternatively premixes of the silicone resin and/or the silicone gum may be prepared prior to incorporation into the final blending step to facilitate silicone dissolution and ease of manufacturing. Depending on the formula composition, the order of ingredient addition may also vary such as the addition of the

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# United States Patent [19]

Hill et al.

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[54] **ULTRAMULSION BASED ORAL CARE COMPOSITIONS**

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[52] U.S. Cl. ..... 424/49; 132/321; 132/323; 424/401; 433/216; 433/217.1

[58] Field of Search ..... 424/401, 49; 132/321, 132/323; 433/216, 217.1

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*Primary Examiner*—Teresa Mosley

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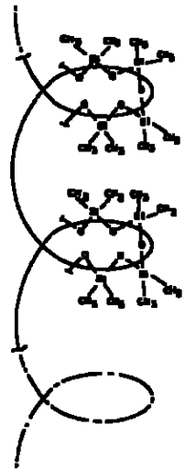
[57] ABSTRACT

The present invention relates to various oral care products containing, stable, dispersions of certain high viscosity silicones in certain surfactants; wherein:

a. the dispersed silicones, which are insoluble in said surfactant, are oriented by the surfactant such that when dispersed in water they are particularly adept at forming oriented, coatings on surfaces of the mouth with enhanced substantivity, and

b. the particle size of the dispersed silicone is from between about 0.1 and about 10 microns, with a particle size distribution such that from between about 80 and 95% of the dispersed silicone is within this particle size range. These stable dispersions are described as ULTRAMULSIONS, which, together with their physical properties, when contained in oral care products, provide these oral care products with enhanced substantivity to mouth surfaces, where the non continuous silicone phase functions as a reservoir for various active ingredients contained therein.

30 Claims, 4 Drawing Sheets



of emulsions with certain features of microemulsions. That is, like emulsions, they are two phase systems comprising a silicone dispersed in a continuous, surfactant phase, wherein the silicone is insoluble in the surfactant. Unlike emulsions, but like microemulsions, these dispersions are stable. Unlike microemulsions, but like emulsions, mechanical work is required to form ultraemulsions. Unlike microemulsions, but like emulsions, these ULTRAMULSION™ dispersions are not formed spontaneously. Like emulsions, the ULTRAMULSION™ dispersions do not contain a cosolvent commonly found in microemulsions. Of course, the ULTRAMULSION™ dispersions of the present invention can be dispersed in various liquids such as water as stable dispersions. These ULTRAMULSION™ dispersions have excellent utility in various oral care products. See various examples below and the various tables below.

While not wishing to be bound by theory, it is hypothesized that unlike either emulsions or microemulsions, the dispersed silicones of the ULTRAMULSION™ dispersions of the present invention are uniquely oriented with their polar moieties in one general plane and their hydrophilic moieties in a plane approximately opposite that of the polar moieties. This orientation promotes stability as well as bonding between the polar or hydrophilic moieties and various surfaces in the oral cavity thereby effecting oriented, monolayer coatings of the silicone onto these surfaces. These oriented dispersions of silicones have a surprising broad range of utility in oral care as detailed in the various examples below. This orienting is illustrated in FIGS. 1 & 2.

The emulsifying effects of uncoiling of the silicone molecule with the oxygen moieties generally oriented in one plane distinct from that of the organo moieties as illustrated in FIGS. 1 and 2, are further substantiated by the following references: *Eur. Poly. J.*, 26: 654 (1990); *J. Chem. Phys.*, 49: 1398 (1965); *J. Chem. Phys.*, 54: 5011 (1971); *J. Chem. Phys.*, 59: 3825 (1973); *Macromolecules*, 7: 229 (1974); *Macromolecules*, 11: 627 (1978) and "Rubber-Like Elasticity: A Molecular Primer," J. Mark, New York, Wiley-Interscience, 1988.

Methods of preparing polyorganosiloxane emulsions with an average particle size of less than about 0.3 microns and polyorganosiloxane microemulsions with an average particle size of less than about 0.14 micron are described in U.S. Pat. No. 4,620,878. Preparation of oil-in-water microemulsions are described in U.S. Pat. No. 4,146,499. Specific surface active compositions used as emulsifiers with diorganopolysiloxanes to form transparent microemulsions are described in U.S. Pat. Nos. 4,0562.331 and 3,975.294. U.S. Pat. No. 3.433,780 teaches the preparation of colloid silane suspensions. See also "Chemistry and Technology of Silicones," W. Noll, pp. 428 to 431 (1968); *Journal of Society of Cosmetic Chemists*, 25: 609–619 (1974) and *Journal of Colloid & Interface Science*, 44: 242–248 (1973).

Micellar dispersions, microemulsions, transparent emulsions are described in detail in "Annals of the New York Academy of Science," Shulman & Montagne (1961); U.S. Pat. No. 2,356.205, "The Theory of Emulsions & Their Technical Treatment," 5th Edition, 1954, U.S. Pat. Nos. 3.497.006; 3.506.070, 3,254.714 and 3.307.628.

The aqueous-free ULTRAMULSION™ dispersions of silicones in surfactants as described herein are neither taught nor suggested by the foregoing references.

## BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 illustrates the "coiled" molecular configuration proposed to polydimethylsiloxanes;

FIG. 2 illustrates the proposed molecular configuration of oriented polydimethylsiloxanes after ULTRAMULSION™ dispersion processing;

FIG. 3 illustrates schematically an ULTRAMULSION™ dispersion process of the invention;

FIGS. 4 and 5 illustrate that the ULTRAMULSION™ dispersions of the invention produced via various high shear dispersing means having particle size distribution of 80+% under 10 microns; and

FIGS. 6 and 7 are a graph and a chart respectively showing the effect of increased silicone viscosity on plaque building in a certain oral care product.

## DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring to the drawings, FIG. 1 illustrates the accepted "coiled" configuration advanced for polydimethylsiloxanes, wherein the methyl moieties are oriented outward while the oxygen moieties are oriented inward towards the axis of the coil or helix. This configuration does not readily promote "bonding" between the oxygen moieties and compatible surfaces such as those in the oral cavity.

FIG. 2 illustrates the "uncoiled oriented" configuration proposed for polydimethylsiloxanes that have been dispersed in the stable, ULTRAMULSION™ dispersion of the present invention, wherein the oxygen moieties are generally oriented in one plane distinct from that of the methyl moieties. This proposed uncoiled oriented configuration appears to support the unique and unexpected stability "bonding and enhanced substantivity" properties of the ULTRAMULSION™ dispersion of the present invention, as evidenced by the various coating applications of these ULTRAMULSION™ dispersions to surfaces in the oral cavity. See examples below.

FIG. 3 illustrates the ULTRAMULSION™ dispersion process of the present invention wherein a nonionic surfactant and a polydimethylsiloxane 1, substantially free from water and co-solvent, are mixed in vessel 2, provided with mixing means 3, heat source 4, and inert head space 5. The heated and mixed surfactant and poly-dimethylsiloxane 6, is then subjected to high shear dispersion at an elevated temperature in dispersing means 7, to produce the ULTRAMULSION™ dispersion 8, of the invention.

FIG. 4 is a chart describing the particle size distribution of an ULTRAMULSION™ dispersion of the invention containing 95–5% by weight nonionic surfactant and 5–50% by weight polydimethylsiloxane (2.5 million cs) produced in a continuous process with an IKA Work dispersing means, (high shear dispersing) with an inlet temperature of 140° C. and an outlet temperature of 210° C.

FIG. 5 is a chart describing the particle size distribution of an ULTRAMULSION™ dispersion of the invention containing 95–50% by weight nonionic surfactant and 5–50% by weight polydimethylsiloxane (2.5 million cs) produced in a batch process with a Ross M/E 100 LC dispersing means fitted with a 20 mesh screen, operated at a temperature from 120° to 160° C.

FIGS. 6 and 7 disclose the influence of increasing viscosity of the silicone in the ULTRAMULSION™ dispersion on the anti-plaque effect of this ULTRAMULSION™ dispersion when introduced into the oral cavity several times throughout the day as a mint. This reduction in plaque buildup is a substantial advance in establishing the efficacy and value of away-from-home oral care products such as mints.

and/or a substantial portion of the dispersed phase resists separation. This latter definition is particularly applicable to higher viscosity silicones. See Table 2.

b. water-free means, that the ULTRAMULSION™ dispersion of silicone and surfactant is substantially free from water.

c. solvent free means, that the ULTRAMULSION™ dispersion of silicone and surfactant is substantially free from cosolvents such as ethanol, isopropanol, etc.

d. oriented means, that the polar moieties of the "uncoiled" polydimethylsiloxane in the ULTRAMULSION™ dispersion are generally aligned in one plane with the hydrophilic oil seeking moieties aligned in a second plane such as illustrated in FIG. 2.

e. monolayer means, that the monomolecular film of the ULTRAMULSION™ dispersion of the invention when dispersed in water is attracted to mucosa and hydroxyapatite by secondary bonding forces to form a substantive coating thereon.

The ULTRAMULSION™ dispersions of the present invention are prepared as follows:

Generally, if not a liquid, the surfactant is heated to a temperature at which it becomes a liquid. The silicone is dispersed in the heated surfactant with various high shear dispersing means.

Specifically the heated surfactant is mechanically stirred along with the silicone to form a pre-emulsion mixture in which the silicone is uniformly dispersed in the surfactant in droplets of a larger size than desired for the ULTRAMULSION™ dispersion but small enough to optimize the subsequent high shear dispersions. This mixture is subjected to high-shear dispersions with a means such as the IKA-WORKS DISPAX-Reactor with at least one superfine generator, alternatively, a Ross Model M.E., 100 LC fitted with a 20 mesh screen or a ultrasonicator such as MEDSONIC XL2010 fitted with 800-C Flow Cell & 800-21CT ¾ inch flanged horn can be used.

Various ULTRAMULSION™ dispersions which are prepared and analyzed are described in detail in the examples below.

TABLE I

| | ORAL CARE % W/W | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Example No. | | | | | | | | | | |
| Component | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 |
| Dimethicone viscosity-centistokes | | | | | | | | | | | |
| 100,000 | 10 | — | — | — | — | — | — | — | — | 33 | — |
| 600,000 | — | 10 | — | — | — | — | 33 | — | — | — | — |
| 2,500,000 | — | — | 10 | — | — | — | — | 33 | — | — | 10 |
| 4,000,000 | — | — | — | 10 | — | — | — | — | 33 | — | — |
| 30,000,000 | — | — | — | — | 10 | — | — | — | — | — | — |
| 50,000,000 | — | — | — | — | — | 10 | — | — | — | — | — |
| Poloxamer - 188 | — | — | — | — | — | — | — | — | — | 67 | — |
| Poloxamer - 288 | — | — | — | — | — | — | — | — | — | — | 90 |
| Poloxamer - 338 | 90 | 90 | 90 | 90 | 90 | 90 | — | — | — | — | — |
| Poloxamer - 407 | — | — | — | — | — | — | 67 | 67 | 67 | — | — |

Specific poloxamer/polydimethylsiloxane ULTRAMULSION™ dispersions suitable for use with various oral care products were prepared and analyzed as described in Table 2 below:

TABLE 2

| | ORAL CARE % W/W | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Example No. | | | | | | | | | |
| | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 | 21 |
| Component | | | | | | | | | | |
| Dimethicone viscosity-centistokes | | | | | | | | | | |
| 600,000 | — | 11.6 | — | — | — | 10.0 | — | — | — | — |
| 2,500,000 | 10.0 | — | — | 11.9 | 11.9 | — | — | — | — | 14.0 |
| 4,000,000 | — | 11.6 | — | — | — | — | — | — | — | — |
| 30,000,000 | — | — | — | — | — | 11.6 | — | — | — | — |
| 50,000,000 | — | — | — | — | — | — | 11.6 | 10.0 | — | — |
| Lipid Soluble | | | | | | | | | | |
| Component | | | | | | | | | | |
| Mixture Of: | | | | | | | | | | |
| Thymol - 24% | — | — | — | — | — | — | — | — | — | — |
| Menthol - 16% | 10.0 | — | — | — | — | — | — | — | — | — |
| Eucalyptol - 36% | — | — | — | — | — | — | — | — | — | — |
| Methyl Salicylate - 24% | — | — | — | — | — | — | — | — | — | — |
| Stannous Fluoride | — | — | — | — | 1.75 | — | — | — | — | — |
| Triclosan | — | 1.16 | 1.16 | — | — | — | 1.16 | 1.16 | — | 2.0 |
| Chlorhexidine | — | — | — | — | — | — | — | — | — | — |
| Metronidazole | — | — | — | 1.3 | — | — | — | — | — | — |
| Benzocaine | — | — | — | — | 1.0 | — | — | — | — | — |
| Surfactant Poloxamer 338 | 90.0 | 97.24 | 97.24 | 86.8 | 87.25 | 88.0 | 87.24 | 87.24 | 90.0 | 84.0 |

dispersion provides the base functioning as a carrier for specific active ingredients such as Candida yeast sp. antifungal agents such as stannous fluoride.

The present invention has been described in detail, including the preferred embodiments thereof. However, it will be appreciated that those skilled in the art, upon consideration of the present disclosure, may make modifications and/or improvements on this invention and still be within the scope and spirit of this invention as set forth in the following claims.

What is claimed is:

1. An oral care composition selected from the group consisting of rinses, sprays, gels, creams, toothpastes, tooth powders, dental floss, interproximal simulators, mists and chewing gum, wherein said composition contains an aqueous-free high shear or ULTRAMULSION™ dispersion, formed by heating a mixture of surfactant and silicone, followed by high shear mixing wherein:

a. the silicone is insoluble in said surfactant, has a viscosity greater than about 100,000 cs; and a particle size up to about 10 microns;

b. the surfactant to silicone ratio in the ULTRAMULSION dispersion is from between about 400:1 and about 1:1; and the surfactant has an orienting effect on the silicone,

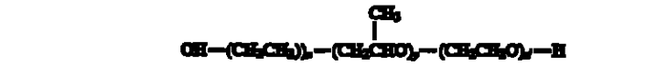
c. the ULTRAMULSION dispersion forms stable dispersions in aqueous containing oral care compositions, and

d. said oral care composition exhibits enhanced substantivity to surfaces in the oral cavity while the dispersed silicone phase of said ULTRAMULSION dispersion functions as a reservoir for one or more lipid soluble and lipid dispersible oral care active ingredients.

2. An oral care composition according to claim 1, wherein said ULTRAMULSION dispersion comprises a nonionic poloxamer surfactant and polydimethylsiloxane wherein:

a. said polydimethylsiloxane has the chemical composition $(CH_3)_3SiO[SiO(CH_3)_2]_nSi(CH_3)_3$, wherein n is a whole number;

b. said surfactant has the chemical composition



wherein x, y, and x' are whole numbers;

c. the viscosity of the polydimethylsiloxane ranges from between about 2.5 million and about 50 million cs;

d. the particle size of most of the polydimethylsiloxane in the ULTRAMULSION dispersion is from between about 0.1 and about 10 microns;

e. from between about 80% and 95% of said polydimethylsiloxane particles in the ULTRAMULSION dispersion are from between about 1 and about 10 microns;

f. the nonionic surfactant is a polyoxyethylene-polyoxypropylene block copolymer having a molecular weight from between about 1,100 and about 150,000;

g. the ratio of surfactant to polydimethylsiloxane is from between about 400:1 and about 1:2; and

h. the ULTRAMULSION dispersion dispersed in water based oral care composition is stable.

3. A method of manufacturing ULTRAMULSION™ dispersions suitable for oral care compositions said method comprising, heating said surfactant and silicone mixture in a heated, stirred vessel substantially free from water, followed by subjecting said mixture to high shear dispersion; wherein;

a. the silicone is insoluble in said surfactant, has a viscosity ranging from about 100,000 cs up to about 50 million cs, and a particle size up to about 10 microns,

b. the surfactant to silicone ratio in the high shear dispersion is from between about 400:1 and about 1:1; and the surfactant has an orienting effect on the silicone,

c. the silicone is oriented, exhibits enhanced substantivity to surfaces in the oral cavity and functions as a reservoir for one or more lipid soluble and lipid dispersible hair care active ingredients.

4. A method according to claim 3, wherein the heated vessel is provided with an inert head of gas.

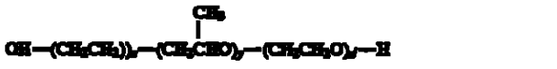
5. A method according to claim 3, wherein said high shear dispersing means is fitted with a small orifice.

6. A method according to claim 3 wherein said high shear dispersing means is an ultrasonication means.

7. A stable aqueous based oral care composition containing a dispersed therein an ULTRAMULSION dispersion comprising a nonionic poloxamer surfactant and a polydimethylsiloxane insoluble in said surfactant wherein:

a. said polydimethylsiloxane has the chemical composition $(CH_3)_3SiO[SiO(CH_3)_2]_nSi(CH_3)_3$, wherein n is a whole number;

b. said surfactant has the chemical composition



wherein x, y, and x' are whole numbers;

c. the viscosity of the polydimethylsiloxane ranges from between about 2.5 million and about 50 million cs;

d. the particle size of most of the polydimethylsiloxane in the ULTRAMULSION dispersion is from between about 0.1 and about 10 microns;

e. from between about 80% and 95% of said polydimethylsiloxane particles in the ULTRAMULSION dispersions are from between about 1 and about 10 microns;

f. the nonionic surfactant is a polyoxyethylene-polyoxypropylene block copolymer having a molecular weight from between about 1,100 and about 150,000;

g. the ratio of surfactant to polydimethylsiloxane is from between about 400:1 and about 1:2; and

h. the ULTRAMULSION dispersion dispersed in water is stable.

8. An oral care composition according to claim 7, wherein the ratio of said surfactant to said silicone is 9:1 and 90% of the silicone particles are from between about 1 and 3 microns.

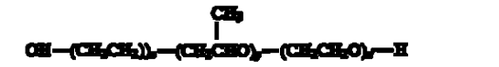
9. An oral care composition according to claim 7, wherein the ratio of said surfactant to said silicone is 2:1 and 100% of the silicone dispersion is less than 10 microns.

10. An oral care composition according to claim 7, wherein the ratio of said surfactant to said silicone is 1:1 and the silicone particles in said ULTRAMULSION dispersion are less than 10 microns.

11. An aqueous based rinse composition containing an ULTRAMULSION dispersion comprising a nonionic poloxamer surfactant and polydimethylsiloxane insoluble in said surfactant wherein:

a. said polydimethylsiloxane has the chemical composition $(CH_3)_3SiO[SiO(CH_3)_2]_nSi(CH_3)_3$, wherein n is a whole number;

b. said surfactant has the chemical composition



wherein x, y, and x' are whole numbers;

c. the viscosity of the polydimethylsiloxane ranges from between about 100,000 and about 4 million cs;

d. the particle size of most of the polydimethylsiloxane in the ULTRAMULSION dispersion is from between about 0.1 and about 10 microns;

e. from between about 80% and 95% of said polydimethylsiloxane particles in the ULTRAMULSION dispersion are from between about 1 and about 10 microns;

f. the nonionic surfactant is a polyoxyethylene-polyoxypropylene block copolymer having a molecular weight from between about 1,100 and about 150,000;

g. the ratio of surfactant to polydimethylsiloxane is from between about 400:1 and about 1:2;

h. the ULTRAMULSION dispersion dispersed in water based rinse is stable, and

i. the polydimethylsiloxane contains one or more essential oil active ingredients.

12. An oral care composition according to claim 7, wherein the silicone is a polydimethylsiloxane uncoiled and oriented wherein the oxygen moieties are generally oriented in a plane distinct from that of the methyl/moieties.

13. An oral care composition according to claim 1, wherein the surfactant is selected from the group consisting of, flowable liquids of varying viscosities, pastes, pills and cast solids.

14. A method according to claim 3, wherein the high shear dispersion is achieved with high shear dispersing means selected from the group consisting of superfine dispersion means and ultrasonic dispersion means.

15. An oral care composition according to claim 7, wherein the ratio of surfactant to polydimethylsiloxane is 1:1 and at least 80% of the polydimethylsiloxane dispersed particles are between 1 and 9 microns.

16. An oral care composition according to claim 1, wherein the ratio or surfactant to polydimethylsiloxane is 9:1 and about 90% of the polydimethylsiloxane dispersed particles are between 1 and 3 microns.

17. An oral care composition according to claim 1, wherein the ratio or surfactant to polydimethylsiloxane is 2:1 and about 90% of the polydimethylsiloxane dispersed particles are between 1 and 3 microns.

18. An oral care composition according to claim 7, wherein the ratio of surfactant to polydimethylsiloxane is 4:1 and about 90% of the polydimethylsiloxane dispersed particles are between 1 and 9 microns.

19. An oral care composition according to claim 7, wherein the ratio of surfactant to polydimethylsiloxane is 9.5:0.5 and about 100% of the polydimethylsiloxane dispersed particles are between 1 and 9 microns.

20. An oral care composition according to claim 7, wherein the polydimethylsiloxane has a viscosity of 2.5 million cs and the surfactant is a solid at room temperature.

21. An oral care composition according to claim 1, wherein the silicone contains an active ingredient selected from the group consisting of, anti-plaque, anti-tartar, anti-gingivitis and anti-periodontitis active ingredients.

22. An oral care composition according to claim 21, wherein the silicone contains triclosan.

23. An oral care composition according to claim 21, wherein the silicone contains a mixture of essential oils selected from the group consisting of thymol, eucalyptol, menthol and methyl salicylate.

24. An oral care composition according to claim 21, wherein the silicone contains stannous fluoride.

25. An oral care composition according to claim 21, wherein the silicone contains chlorhexidine.

26. An oral care composition according to claim 21, wherein the silicone contains metronidazole.

27. An oral care composition according to claim 1, wherein the composition is a gel for treating periodontal pockets.

28. An oral care composition according to claim 1, wherein the composition is a toothpaste containing triclosan in said silicone.

29. An oral care composition according to claim 1, wherein the composition is a dental floss where the silicone contains one or more antimicrobials selected from the group consisting of stannous fluoride, triclosan, chlorhexidine and metronidazole.

30. An oral care composition according to claim 1, wherein the composition is a gel and the silicone contains benzocaine.

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# United States Patent [19]

**Watson**

[11] **3,934,001**

[45] **Jan. 20, 1976**

[54] **ORAL COMPOSITIONS CONTAINING GERMICIDALLY ACTIVE PLASTIC POWDERS**

[75] Inventor: **Charles Andrew Watson, Ruislip, England**

[73] Assignee: **Lever Brothers Company, New York, N.Y.**

[22] Filed: **July 31, 1970**

[21] Appl. No.: **64,126**

**Related U.S. Application Data**

[63] **Continuation of Ser. No. 836,217, June 12, 1969, abandoned, which is a continuation of Ser. No. 599,692, Dec. 7, 1966, abandoned.**

[30] **Foreign Application Priority Data**

**Dec. 7, 1965 United Kingdom ..... 51752/65**

[52] **U.S. Cl. ..... 424/49; 424/78; 424/81; 424/83**

[51] **Int. Cl.² ..... A61A 7/16**

[58] **Field of Search ..... 424/49–58**

[56] **References Cited**

**FOREIGN PATENTS OR APPLICATIONS**

**1,504,155 10/1967 France ..... 424/49**

**OTHER PUBLICATIONS**

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**Dubos et al, Bacterial and Mycotic Infections of Man, 2nd ed., published by J. B. Lippincott Co., Phila., 1952, p. 663.**

**Simonds-Church, The Encyclopedia of Basic Materials for Plastics, published by Reinhold Publishing Co., 1967, p. 246.**

**Accepted Dental Therapeutics, 1969/1970, 33rd edition, published by the American Dental Association, Chicago, p. 243.**

**The Washington Sunday Star, July 27, 1969, p. B-2.**

*Primary Examiner*—**Richard L. Huff**

*Attorney, Agent, or Firm*—**Arnold Grant, Esq.**

[57] **ABSTRACT**

**Oral compositions are disclosed which contain germicidally active plastic powders. The plastic powders employed are resinous materials having a germicide absorbed therein preferably in a concentration of at least about 0.1% by weight of the resin. Germicides and plastic powders suitable for use in oral compositions are discussed.**

**6 Claims, No Drawings**

# ORAL COMPOSITIONS CONTAINING GERMICIDALLY ACTIVE PLASTIC POWDERS

This application is a continuation of Ser. No. 836,217, filed June 12, 1969, which in turn is a continuation of Ser. No. 599,692, filed Dec. 7, 1966, both now abandoned.

This invention relates to oral compositions.

It has been proposed to include germicides, for example hexachlorophene, in compositions for oral hygiene. Examples of such compositions where inclusion of a germicide has been proposed are dentifrices and mouthwashes. The inclusion of germicides in such compositions produces a beneficial effect in the mouth but it is however limited because the germicide is retained in the oral cavity for only a short time.

It is an object of the present invention to provide a means of producing a longer germicidal effect in the mouth.

It has been found that this can be achieved by including in the oral composition, particles of a water-insoluble material which have been pretreated with a germicide so that they are impregnated with the germicide. The longer-lasting germicidal effect produced by oral compositions incorporating such germicide-containing particles is believed to be due to the trapping of some of the particles in crevices in the mouth and release of germicide from such particles.

Accordingly, the present invention provides an oral composition comprising particles of a water-insoluble material which have been impregnated with a germicide.

The water-insoluble material should, of course, be non-toxic, the particles are preferably white or nearly white. Suitable as the particles are plastic materials, especially thermoplastic resins. Preferred plastics materials are polyvinylchloride, polyethylene, polypropylene, polymethylmethacrylate, and copolymers of polyvinylchlorides and polyvinylalcohol. The particles desirably have a size less than 50 microns. Especially suitable are particles having a size within the range 0.1 to 20 microns. While hexachlorophene is the preferred germicide that is used, other germicides known in the art as suitable for use in oral hygiene may be used, for example chlorhexidene, tyrothricin and quaternary ammonium germicides.

The particles of the water-insoluble material may be impregnated with the germicide by pretreating them with a solution of the germicide in a solvent capable of being absorbed by the particles whereby the germicide diffuses into and is absorbed by the particles. To increase the rate of diffusion of the solvent and germicide into the particles the treatment is preferably carried out at an elevated temperature.

The oral composition may be in the form of a dentifrice or in the form of a mouthwash or other product for the care of the oral cavity. If the oral composition is a dentifrice this may be in the form of a liquid, paste, powder, tablets or solid block. The amount of the germicide containing particles in the oral preparation will be chosen to suit the particular form of the product. In a toothpaste dentifrice for example, suitable amounts of the germicide-containing particles are from 1 to 50% by weight, preferably 3 to 35% by weight. The amount used in the dentifrice or other oral composition will also be chosen having regard to the amount of the germicide in the particles.



The amount of the germicide in the particles is preferably at least 0.05% by weight of the oral composition.

The oral composition will also comprise the usual ingredients. Thus, toothpastes, for instance, will usually comprise abrasive material, humectant, binder, foaming agent, sweetening agent and flavouring agent, and may also contain germicide other than that in the particles.

It is believed that the germicide-containing water-insoluble particles employed in the above oral compositions are themselves novel, and in accordance with another aspect of the present invention, the invention also relates to such germicide-containing particles themselves. The particles preferably contain at least 0.1% by weight of absorbed germicide, preferably 1% or more of germicide.

The invention also relates to a method of making an oral composition comprising the steps of contacting particles of a water-insoluble material with a germicide in solution in a solvent capable of being absorbed by the particles, separating, if desired, the particles containing absorbed solvent and germicide from any excess solvent and germicide, and including the germicide-containing particles in an oral composition.

The following Examples illustrate the invention. Parts are by weight.

## EXAMPLE 1

1 part of hexachlorophene was dissolved in a mixture of 5 parts of propylene glycol and 20 parts of glycerine. 10 parts of polyvinylchloride powder (particle size in the range 1 to 20 microns) were then added. The mixture was stirred at 90° to 100°C. for 1 to 2 hours. The mixture was then cooled and the powdered plastics material filtered off, washed with hot propylene glycol followed by distilled water and dried in an oven at 50°C. The content of hexachlorophene in the particle was determined and found to be 6% by weight. The particles were then included in a dentifrice.

A typical composition of a dentifrice containing the polyvinylchloride particles is the following:

| Ingredient | Parts |
|---|---|
| Plastics material impregnated with germicide | 30.0 |
| Polishing Agent | 12.5 |
| Humectant | 18.0 |
| Binding Agent | 1.6 |
| Foaming Agent | 1.5 |
| Sweetening Agent | 0.2 |
| Flavouring Agent | 1.0 |
| Water | to 100.0 |

## EXAMPLE 2

5 parts of hexachlorophene were dissolved in a mixture of 20 parts cineole and 18 parts propylene glycol. 50 parts of polyethylene powder (particle size in the range 1 to 20 microns) were added and the mixture heated at 70°C. for three hours with stirring. The powdered polyethylene was then filtered off, washed with hot propylene glycol followed by distilled water and dried in an oven at 50°C. The amount of hexachlorophene introduced in the polyethylene particles was 1.25% by weight.

A typical composition of a dentifrice comprising the germicide-containing polyethylene powder produced as above is the following:

| Ingredient | Parts |
|---|---|
| Polyethylene powder impregnated with germicide | 25.0 |
| Polishing Agent | 12.5 |
| Humectant | 20.0 |
| Foaming Agent | 1.2 |
| Binding Agent | 1.4 |
| Sweetening Agent | 0.2 |
| Flavouring Agent | 1.0 |
| Water | to 100.0 |

Toothpastes having the compositions indicated in Examples 1 and 2 were stored at 37°C. for times as indicated in the Table below. The plastics materials were then separated from the toothpastes and the level of hexachlorophene in them determined. The results are indicated in the Table.

TABLE

| | Content of Hexachlorophene (after time indicated) | | | | | |
|---|---|---|---|---|---|---|
| | Initially | 1 day | 3 days | 7 days | 17 days | 63 days |
| Ex. 1 | 6.0 | 5.6 | 5.3 | — | 4.6 | 4.6 |
| Ex. 2 | 1.25 | 0.88 | 0.34 | 0.32 | — | — |

The results show that an equilibrium is reached between the germicide in the polymer and the germicide in the aqueous toothpaste phase.

EXAMPLE 3

2.5 parts of hexachlorophene were dissolved in 100 parts of propylene glycol. 25 parts of polymethylmethacrylate powder (particle size in the range 1 to 20 microns) were added and the mixture heated at 90°C. for three hours. The polymethylmethacrylate particles were then filtered off, washed with hot propylene glycol followed by distilled water and dried in an oven at 50°C. The amount of hexachlorophene introduced in the plastics particles was 0.25% by weight.

The plastics particles containing hexachlorophene may be included in a toothpaste of the composition described in Example 1 or Example 2.

EXAMPLE 4

1 part of hexachlorophene was dissolved in a mixture of 12.5 parts of propylene glycol and 12.5 parts of glycerine. 10 parts of a powdered copolymer of polyvinylchloride and (10%) polyvinyl alcohol (particle size in the range 1 to 20 microns) were added. The mixture was stirred at 90°C. for three hours. the powdered plastics material was then filtered off, washed with hot propylene glycol followed by distilled water and dried at 50°C. The amount of hexachlorophene contained in the polymer was about 2.4% by weight.

The plastics particles containing hexachlorophene may be included in a toothpaste of the composition described in Example 1 or Example 2.

EXAMPLE 5

The following is an example of typical mouthwash preparation in accordance with the invention.

| Ingredient | Parts |
|---|---|
| Polyvinylchloride impregnated with germicide, prepared as described in Example 1 | 5.00 |
| Ethyl alcohol | 20.00 |
| Flavour | 0.08 |
| Polyoxyethylene stearyl ether | 0.12 |
| Saccharin | 0.02 |
| Water | to 100.00 |

I claim:

1. In a liquid mouthwash preparation having a liquid vehicle and an effective amount of a germicide suitable for use in oral hygiene, the improvement which comprises providing said germicide in the form of non-toxic, water-insoluble thermoplastic resinous particles having a size less than 50 microns, said particles being impregnated with said germicide suitable for use in oral hygiene in an amount of at least about 0.1 per cent of said particles by weight.

2. A mouthwash preparation in accordance with claim 1 wherein said water-insoluble thermoplastic resinous particles are between 0.1 and 20 microns in diameter.

3. A mouthwash preparation in accordance with claim 1 wherein the amount of said water-insoluble thermoplastic resinous germicide-impregnated particles is sufficient to provide at least 0.05 per cent germicide by weight of the mouthwash composition.

4. A mouthwash preparation in accordance with claim 1 wherein said water-insoluble thermoplastic resinous particles are selected from the group consisting of polyvinylchloride, polyethylene, polypropylene, polymethylmethacrylate and copolymers of polyvinylchloride and polyvinyl alcohol.

5. A mouthwash preparation in accordance with claim 1 wherein said germicide is hexachlorophene.

6. A mouthwash preparation in accordance with claim 1 wherein said water-insoluble thermoplastic resinous germicide impregnated particles contain at least about 1 per cent by weight germicide.

* * * * *

**SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO**

**DIVISION DE NUEVAS CREACIONES**

2020
Bogotá, D.C., Septiembre 6 de 2006

**Abogada**
**JIMENA ESCOBAR URIBE**

| | | |
|---|---|---|
| **ASUNTO** | Radicación No. | 02 035 233 |
| | Trámite | 002 |
| | Evento | 001 |
| | Actuación | 430 |
| | Oficio No. | 10447 |
| | Folios | 0 |

Como resultado del examen de patentabilidad de la solicitud de patente de invención, realizado según lo establece el artículo 45 de la Decisión 486 de la Comisión de la Comunidad Andina, se notifica el siguiente concepto técnico:

**Título de la solicitud:** "Composición para Higiene Oral".

**1. Documentos estudiados**

Para el presente estudio se tuvieron en cuenta el capítulo descriptivo comprendido en los folios 7 - 21 y el capítulo reivindicatorio de los folios 22 – 23 de la solicitud.

**2. Objeto de la invención**

El objeto de la presente solicitud de patente de invención consiste en:

Las reivindicaciones 1 – 6 se refieren a una composición para higiene oral que contiene:

- Vehículo acuoso
- Agente activo germicida (Triclosán) adsorbido en partículas de
- Polímero insoluble en agua (polisiloxano) de 80 – 100 nm.
- Opcionalmente, contiene: Alcohol etílico (antiséptico), pluronic (tensioactivo), metilparabeno, propilparabeno (conservantes), fluoruro de sodio (anticaries), fosfato de sodio (regulador de pH), sacarosa (edulcorante), cloruro de cetil piridinio (antiséptico), menta (saborizante), blue n-1 (colorante).

El problema planteado en esta solicitud es la necesidad de una composición para higiene oral con acción germicida eficiente y prolongada.

Para resolver este problema técnico la solicitud en estudio proporciona una composición para higiene oral que contiene agente activo germicida absorbido en partículas de polímero, en un vehículo acuoso.

El objeto de la invención definido anteriormente se clasificó de acuerdo con la IPC: A 61 K 7/16.

También se reivindica prioridad de la Solicitud de Patente de Brasil PI 0101911-2 de abril 30 de 2001, con respecto a la cual la fecha de presentación de la actual solicitud cumple el plazo máximo permitido.

### 3. Determinación del Estado de la Técnica

El artículo 16 de la decisión 486 de la comisión de la Comunidad Andina que establece que "*El estado de la técnica comprenderá todo lo que haya sido accesible al publico por una descripción escrita u oral, utilización, comercialización o cualquier otro medio antes de la fecha de presentación de la solicitud de patente o, en su caso, de la prioridad reconocida.*

*Solo para el efecto de la determinación de la novedad, también se considerará dentro del estado de la técnica, el contenido de una solicitud de patente en trámite ante la oficina nacional competente, cuya fecha de presentación o de prioridad fuese anterior a la fecha de presentación o de prioridad de la solicitud de patente que se estuviese examinando, siempre que dicho contenido esté incluido en la solicitud de fecha anterior cuando ella se publique o hubiese transcurrido el plazo previsto en el artículo 40*".

Considerando que la fecha de presentación de la primera solicitud de patente de la cual se reivindica prioridad es abril 30 de 2001, se consultaron las bases de datos y los archivos con que cuenta la entidad con fecha de publicación anterior y se encontraron los siguientes documentos que anticipan el objeto de esta solicitud:

| No. | Documento | Título | Fecha de Publicación |
|---|---|---|---|
| D1 | WO 01 01942 | Systems Comprising Organosiloxane Resins For Delivering Oral Care Substances And For Prolonging Such Delivery | Ene 11, 2001 |
| D2 | US 5 651 959 | Ultramulsion based oral care compositions | Jul 29, 1997 |
| D3 | US 3 934 001 | Oral compositions containing germicidally active plastic powders | Ene 20, 1976 |

### 4. Evaluación de los requisitos de Patentabilidad

El artículo 14 de la Decisión 486 de la Comisión de la Comunidad Andina establece que "*Los Países Miembros otorgarán patentes para las invenciones, sean de producto o de procedimiento, en todos los campos de la tecnología, siempre que sean nuevas, tengan nivel inventivo y sean susceptibles de aplicación industrial*".

En cumplimiento del artículo 14 de la Decisión 486, se procedió a evaluar el cumplimiento de los requisitos de novedad, nivel inventivo y aplicación industrial, con el fin de establecer la patentabilidad de la invención reivindicada en la solicitud en estudio.

#### Evaluación de la Novedad

El Art. 16 de la Decisión 486 de la Comisión de la Comunidad Andina establece: "*Una invención se considerará nueva cuando no está comprendida en el estado de la técnica. El estado de la técnica comprenderá todo lo que haya sido accesible al público por una descripción escrita u oral, utilización comercialización o cualquier otro medio antes de la fecha de presentación de la solicitud de patente, o, en su caso, de la prioridad*

*reconocida.*

*Solo para el efecto de la determinación de la novedad, también se considerará dentro del estado de la técnica, el contenido de una solicitud de patente en trámite ante la oficina nacional competente, cuya fecha de presentación o de prioridad fuese anterior a la fecha de presentación o de prioridad de la solicitud de patente que se estuviese examinando, siempre que dicho contenido esté incluido en la solicitud de fecha anterior cuando ella se publique o hubiese transcurrido el plazo previsto en el Art 40".*

Las reivindicaciones 1 – 6 se refieren a una composición para higiene oral que contiene:
- Vehículo acuoso
- Agente activo germicida (Triclosán) absorbido en partículas de
- Polímero insoluble en agua (polisiloxano) de 80 – 100 nm.
- Opcionalmente, contiene: Alcohol etílico (antiséptico), pluronic (tensioactivo), metilparabeno, propilparabeno (conservantes), fluoruro de sodio (anticaries), fosfato de sodio (regulador de pH), sacarosa (edulcorante), cloruro de cetil piridinio (antiséptico), menta (saborizante), blue n-1 (colorante).

D1 enseña (ver págs 1, 4, 13) una composición para higiene oral que contiene: vehículo acuoso, agente activo germicida (Triclosán), resina de organosiloxano. Esta composición tiene efecto germicida más prolongado.

D2 enseña (ver cols 23, 24, 25, 26) una composición para higiene oral que contiene: vehículo acuoso y silicona que contiene agente activo germicida (Triclosán). Esta composición tiene efecto germicida más prolongado.

D3 enseña (ver cols 1 – 4) una composición para higiene oral que contiene: vehículo acuoso y agente activo germicida absorbido en partículas de resina termoplástica. Esta composición tiene efecto germicida más prolongado.

Se observa que los elementos característicos de las composiciones reivindicadas, es decir el agente activo germicida adsorbido en partículas de polímero, en un vehículo acuoso, ya era conocido en D1, D2 y D3; por lo cual las composiciones de las reivindicaciones 1 – 6 no pueden considerarse nuevas.

**Evaluación del Nivel Inventivo**

El artículo 18 de la Decisión 486 de la Comisión de la Comunidad Andina establece que *Se considerará que una invención tiene nivel inventivo, si para una persona del oficio normalmente versada en la materia técnica correspondiente, esa invención no hubiese resultado obvia ni se hubiese derivado de manera evidente del estado de la técnica.*

Las reivindicaciones 1 – 6 se refieren a una composición para higiene oral que contiene:
- Vehículo acuoso
- Agente activo germicida (Triclosán) absorbido en partículas de
- Polímero insoluble en agua (polisiloxano) de 80 – 100 nm.
- Opcionalmente, contiene: Alcohol etílico (antiséptico), pluronic (tensioactivo), metilparabeno, propilparabeno (conservantes), fluoruro de sodio (anticaries), fosfato de sodio (regulador de pH), sacarosa (edulcorante), cloruro de cetil piridinio (antiséptico), menta (saborizante), blue n-1 (colorante).

D1 enseña (ver págs 1, 4, 13) una composición para higiene oral que contiene: vehículo acuoso, agente activo germicida (Triclosán), resina de organosiloxano. Esta composición tiene efecto germicida más prolongado.

D2 enseña (ver cols 23, 24, 25, 26) una composición para higiene oral que contiene: vehículo acuoso y silicona que contiene agente activo germicida (Triclosán). Esta composición tiene efecto germicida más prolongado.

D3 enseña (ver cols 1 – 4) una composición para higiene oral que contiene: vehículo acuoso y agente activo germicida absorbido en partículas de resina termoplástica. Esta composición tiene efecto germicida más prolongado.

Se observa que los elementos característicos de las composiciones reivindicadas, es decir el agente activo germicida adsorbido en partículas de polímero, en un vehículo acuoso, ya era conocido en D1, D2 y D3; por lo cual las composiciones de las reivindicaciones 1 – 6 no pueden considerarse nuevas.

Se observa que:

(a) debido a que los elementos que conforman la composición para higiene oral reivindicada, es decir el vehículo acuoso y el agente activo germicida (Triclosán) absorbido en partículas de polímero insoluble en agua (polisiloxano); eran conocidos en las anterioridades; una persona de habilidades ordinarias en el arte adaptaría fácilmente estos elementos a la preparación de la composición de esta solicitud;
(b) las reivindicaciones de esta solicitud no mencionan una característica que conceda un efecto diferente o especial a la composición para higiene oral reivindicada, que pueda considerarse como un aporte a la técnica, así que es obvia para una persona versada en la materia;
(c) el problema planteado en esta solicitud que era la necesidad de una composición para higiene oral con acción germicida eficiente y prolongada se resuelve proporcionando una composición para higiene oral que contiene agente activo germicida absorbido en partículas de polímero, en un vehículo acuoso, es decir de la misma manera como se resolvía en las anterioridades;
(d) el resultado obtenido: es decir la composición para higiene oral que contiene agente activo germicida absorbido en partículas de polímero, en un vehículo acuoso, no es inesperado;
(e) no parece haberse superado un prejuicio técnico anterior, con la composición para higiene oral (que contiene agente activo germicida absorbido en partículas de polímero, en un vehículo acuoso) propuesta

de manera que el objeto reivindicado no tiene nivel inventivo.

## 5. Conclusión

En conclusión, los requisitos de patentabilidad de la presente solicitud están afectados así:

| Item | Documento | Reivindicaciones afectadas | Requisito que afecta |
|---|---|---|---|
| D1 | WO 01 01942 | 1 – 6 | Novedad y Nivel inventivo |
| D2 | US 5 651 959 | 1 – 6 | Novedad y Nivel inventivo |
| D3 | US 3 934 001 | 1 – 6 | Novedad y Nivel inventivo |

En cumplimiento del artículo 45 de la Decisión 486 de la Comisión de la Comunidad Andina, se le indica que debe dar respuesta dentro del término de sesenta días contados a partir de la fecha de la notificación de la presente comunicación. En caso de no dar respuesta al presente requerimiento o si a pesar de la respuesta subsistieran los impedimentos para la concesión, se denegará la patente.

Notifíquese el presente oficio conforme a lo dispuesto en el numeral 5.2, literal e), del capítulo quinto del título primero de la Circular Unica No. 10 de 2001.

**ALIX CARMENZA CESPEDES DE VERGEL**
Jefe de la División de Nuevas Creaciones.

El anterior requerimiento fue notificado por fijación en lista, de fecha 14 SET. 2006

| CONCEPTO TÉCNICO<br>No. 1398 | EXAMINADOR TÉCNICO<br>Código 202007 |
|---|---|

# SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

108

RADICACIÓN: 2-35233 EDICTO No. 2606 :

**LA SECRETARÍA GENERAL AD-HOC**
**HACE SABER:**

Que esta Superintendencia profirió Resolución No. 1162 de 29-ENE-2007. Que el contenido de la parte resolutiva es como sigue: El Superintendente de Industria y Comercio.

**RESUELVE**

**ARTICULO PRIMERO:** Negar el privilegio de patente de invención para la solicitud correspondiente a "COMPOSICION PARA HIGIENE BUCAL".

**ARTICULO SEGUNDO:** Notificar personalmente el contenido de la presente resolución a la doctora Jimena Escobar Uribe, o a quien haga sus veces, en su calidad de apoderada de Johnson & Johnson Industria e Comercio Ltda., entregándole copia de la misma, advirtiéndole que contra ella procede el recurso de reposición interpuesto ante el Superintendente de Industria y Comercio, al momento de la notificación o dentro de los 5 días hábiles siguientes a ella.

**NOTIFÍQUESE Y CUMPLASE**
Dada en Bogotá, D.C., a los 29 ENE. 2007.

**EL SUPERINTENDENTE DE INDUSTRIA Y COMERCIO,**

**JAIME RIOS ESCOBAR**

Para notificar a los interesados, se fija el presente EDICTO en lugar visible al público en la SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO, por el término de (10) diez días hábiles contados a partir de las 8:00 a.m. de hoy.

Secretaría General Ad-Hoc 13-FEB-2007 Este edicto se desfija hoy 26-FEB-2007 a las 4:30 p.m.
Secretaría General Ad-Hoc
EDICTO No. 2606