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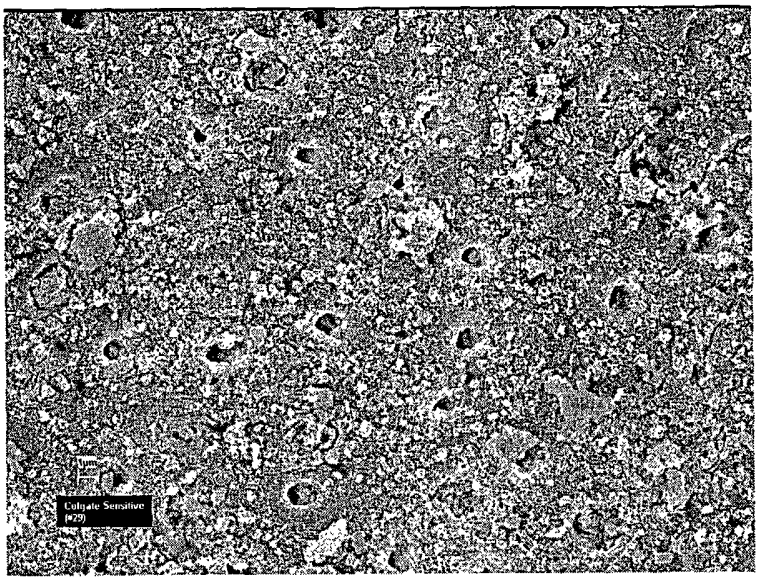
(54) Title: DENTINAL DESENSITIZING DENTIFRICE PROVIDING ENHANCED REMINERALIZATION AND ANTICARIES BENEFITS



(57) Abstract: A dental composition which eliminates or substantially reduces the discomfort and pain associated with dentinal hypersensitivity and exhibits enhanced anticaries and remineralization benefits which composition contains a fluoride ion and a potassium ion releasable salt and has a pH in the range of about 8 to about 9.9, the pH being buffered with phosphate salt.

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DENTINAL DESENSITIZING DENTIFRICE PROVIDING ENHANCED REMINERALIZATION AND ANTICARIES BENEFITS

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to a desensitizing dentifrice composition which eliminates or reduces the discomfort and pain associated with dentinal hypersensitivity and more particularly to a desensitizing dental composition containing a potassium salt desensitizing agents which exhibits unexpected enhanced anticavity and remineralization properties.

2. The Prior Art

Dentinal hypersensitivity is defined as acute, localized tooth pain in response to physical stimulation of the dentine surface as by thermal (hot or cold) osmotic, tactile combination of thermal, osmotic and tactile stimulation of the exposed dentin.

Exposure of the dentine, which is generally due to recession of the gums, or loss of enamel, frequently leads to hypersensitivity. The art has determined that dentine tubules open to the surface have a high correlation with dentine hypersensitivity, Abs, J. Clin. Periodontal. 14,280-4 (1987). Dentinal tubules lead from the pulp to the cementum. When the surface cementum of the tooth root is eroded, the dentinal tubules become exposed to the external environment. The exposed dentinal tubules provide a pathway for transmission of fluid flow to the pulpal nerves, the transmission induced by changes in temperature, pressure and ionic gradients.

It is known to the art that potassium salts are effective in the treatment of dentinal hypersensitivity. For example, U.S. 3,863,006 discloses that toothpastes containing potassium salts such as potassium nitrate desensitize the teeth after tooth brushing for several weeks. It is believed by those skilled in the art that an elevation in the extracellular potassium concentration in the vicinity of pulpal nerves underlying sensitive dentin is responsible for the therapeutic desensitizing effect of topically applied oral products which contain potassium nitrate. Due to passive diffusion of potassium ion into and out of the open dentine tubules, repeated application of the active ingredient is necessary to build up the necessary concentration in the vicinity of the pulpal nerves.

It is believed that the improved pain relief is obtained from the use of potassium salts in combination with gradual mineralization on the dentin surface which can either totally or partially occlude dentin tubules. Total occlusion will dramatically reduce fluid flow within the tubules which stimulates pain. Partial occlusion of the dentin tubules is believed to increase delivery of potassium ion inside the tooth because the inward diffusive flux is less dependent upon tubule radius than outward fluid flow (due to positive pulpal pressures) (See DH Pashley and WG Mathews, Archs. Oral Biol. (1993) **38**, 577-582). Therefore, this enhanced delivery of potassium should enhance relief.

It has also long been known to include fluoride releasing compounds in dentifrices as anticaries agents, and it has been established that these compounds are effective to reduce the incidence of dental caries. Fluoride compounds which are conventionally used are sodium fluoride, sodium monofluorophosphate and stannous fluoride. The fluoride compounds are effective mainly due to the fluoride ions which improve the acid resistance of tooth enamel and accelerate recalcification or remineralization of decayed teeth in their early stage when the demineralization has proceeded only slightly. By remineralization, pre-existing tooth decay and caries can be reduced or eliminated thereby reducing preexisting carious conditions in the tooth structure. The effect of improving the acid resistance of the enamel is believed to be due to the fact that the fluoride ions are incorporated into a crystal lattice of hydroxyapatite which is the main constituent of tooth enamel or, in other words, fluoride ions partially fluoridate hydroxyapatite and simultaneously repair the lattice irregularities.

The effectiveness of fluoride treatment is dependent upon the amount of fluoride ion which is available for deposition on the enamel being treated. It is, therefore, desirable to formulate dentifrice compositions which provide maximum fluoride ion availability in brushing solutions formed using the dentifrice.

While the prior art discloses the use of various oral compositions for the treatment of dentinal hypersensitivity, dental caries, and enamel demineralization there is still a need for additional compositions and methods which provide improved performance in such treatments.

SUMMARY OF THE INVENTION

In accordance with the present invention there is provided an oral composition and method for the treatment of dentinal hypersensitivity which exhibits improved anticavity and remineralization properties, the composition containing a fluoride ion releasing salt and a

potassium releasable salt compound in an orally acceptable vehicle in which the fluoride compound is present at a concentration sufficient to release about 500 to 8800 parts per million (ppm) fluoride, the composition being buffered to maintain an alkaline pH of about 7.5 to about 9 whereby upon repeated application of the composition to the teeth increased relief from dentinal hypersensitivity is experienced by the user accompanied by improved resistance to cavities.

In the Drawings

Fig. 1 is a SEM recorded at 2,000 x magnification, of a dentin disk surface treated with a dual component dentifrice containing high concentrations of a fluoride salt which releases 5000 ppm fluoride ion (1.1% by weight) and potassium nitrate (5% by weight), wherein the first component is buffered to a pH of 6.5 and the second component adjusted to a pH of 9.5 with sodium hydroxide, the pH of the combined components being 7.5 .

Fig.2 is a SEM recorded at 2,000 x magnification, of a dentin disk surface treated with the combined components of a comparative dual component dentifrice of the prior art (US 6,180,089) containing 5% potassium nitrate and a fluoride containing salt which releases 1100 ppm fluoride ion wherein one component is maintained at alkaline pH and the second component maintained at an acid pH, the pH of the combined components being 7.0.

Fig. 3 is a scanning electron photomicrograph (SEM) recorded at 2,000 x magnification of a dentin disk surface treated with a phosphate buffer solution.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The composition of the present invention may be a single phase composition or a dual phase composition.

The dual phase composition is comprised of two components in which a first dentifrice component is maintained at an alkaline pH of about 8.5 to about 9.9 and preferably about 9.0 to about 9.9, and a second dentifrice component is buffered to maintain the pH at a substantially neutral pH level of 6.5 to 7.0. The two components are preferably combined in approximately equal weight proportions, so that about one-half of the concentration of any particular ingredient within either component will be present when the components are combined and applied to the teeth, as by brushing. Both components are preferably formulated to have similar physical characteristics, so that the two components may be

simultaneously delivered in the desired predetermined amounts by extrusion when separately housed in a multicompartmented tube or pump device.

When the dentifrice of the present invention is to be prepared as a single phase product, a buffering agent is incorporated in the dentifrice component which is normally prepared using as a vehicle, one which contains water, humectant, surfactant and an abrasive. The pH of such single component dentifrice is in the alkaline pH buffered in the range of about 7.5 to about 9.0 and preferably about 8.5 to about 9. The buffering agent is preferably an alkali metal phosphate salt and most preferably a mixture of mono- and dibasic sodium phosphate salts. Each phosphate salt is present in the dentifrice at a concentration of about 1.5 to about 5% by weight. The combined amount of buffering agents incorporated in the dentifrice composition is at a concentration of about 5 to about 10% by weight and preferably about 6 to about 10% by weight.

In a dual component dentifrice of the present invention, the one dentifrice component is prepared having an alkaline pH and a composition otherwise similar to that of the other having a buffered neutral pH. The pH of the alkaline component is adjusted to a pH of about 8.5 to about 9.7 and preferably about 9.0 to about 9.5. The pH of the combined dentifrice components is in the range of about 7.5 to about 8.6 and preferably about 7.5 to about 8.5

An alkaline agent such as an alkali metal compound including sodium hydroxide, potassium hydroxide, sodium bicarbonate, sodium carbonate, N-sodium silicate a sodium silicate in 34.6% water available from PQ Corporation) is incorporated in the alkaline pH dentifrice component of the dual component dentifrice in amounts in the range of about 0.5 to about 15% by weight, preferably about 1.0 to about 8% by weight and most preferably at about 1.0 to about 5.0% by weight of the component. Mixtures of the above alkali metal compounds may also be used. Sodium hydroxide is the preferred alkaline agent.

The humectant used in the preparation of the vehicle for the dentifrice composition of the present invention is generally a mixture of humectants, such as glycerol, sorbitol and a polyethylene glycol of molecular weight in the range of 200 to1000, but other mixtures of humectants and single humectants may also be employed. The humectant content is in the range about of 10% to about 50% by weight and preferably about 20 to about 40% by weight of the dentifrice component. The water content is in the range of about 20 to about 50% by weight and preferably about 30 to about 40% by weight.

Thickeners used in the preparation of the dentifrice vehicle include organic and inorganic

thickeners. Inorganic thickeners which may be included in the dentifrice components include amorphous silicas such as Zeodent 165 available from Huber Corporation, and Sylox 15 from W.R. Grace.

Organic thickeners of natural and synthetic gums and colloids may also be used to prepare the dentifrice components of the present invention. Examples of such thickeners are carrageenan (Irish moss), xanthan gum, sodium carboxymethyl cellulose, starch, polyvinylpyrrolidone, hydroxyethylpropylcellulose, hydroxybutyl methyl cellulose, hydroxypropyl methyl cellulose, and hydroxyethyl cellulose.

The inorganic thickener may be incorporated in the dentifrice composition of the present invention at a concentration of about 0.5 to about 5% by weight and preferably about 1 to about 3% by weight. The organic thickener may be incorporated in the compositions of the present invention at a concentration of about 0.1 to about 3% by weight and preferably about 0.4 to about 1.5% by weight.

Surfactants may be incorporated in the dentifrice compositions to provide foaming properties. The surfactant is preferably anionic or nonionic in nature. Suitable examples of anionic surfactants are higher alkyl sulfates such as potassium or sodium lauryl sulfate which is preferred, higher fatty acid monoglyceride monosulfates, such as the salt of the monosulfated monoglyceride of hydrogenated coconut oil fatty acids, alkyl aryl sulfonates such as sodium dodecyl benzene sulfonate, higher fatty sulfoacetates, higher fatty acid esters of 1,2 dihydroxy propane sulfonate.

The surfactant agent is generally present in the dentifrice component composition of the present invention at a concentration of about 0.5 to about 10.0% by weight and preferably about 1.0 to about 5.0% by weight.

Abrasives may be incorporated in the dentifrice composition of the present invention and preferred abrasives are siliceous materials, such as silica. A preferred silica is a precipitated amorphous hydrated silica, such as Sorbosil AC-35, marketed by Crosfield Chemicals, or Zeodent 115 from Huber Company but other abrasives may also be employed, including hydroxyapatite, sodium metaphosphate, potassium metaphosphate, tricalcium phosphate, calcium phosphate dihydrate, anhydrous dicalcium phosphate, calcium pyrophosphate, magnesium orthophosphate, trimagnesium phosphate, calcium carbonate, sodium bicarbonate, alumina trihydrate, aluminum silicate, calcined alumina and bentonite.

The concentration of abrasive in the dentifrice composition of the present invention will normally be in the range of 5 to about 40% by weight and preferably about 10 to 25% by weight.

The source of desensitizing potassium ion is generally a water soluble potassium salt including potassium nitrate, potassium citrate, potassium chloride, potassium bicarbonate and potassium oxalate with potassium nitrate being preferred. The potassium salt is generally incorporated in one or more of the dentifrice components at a concentration of about 1 to about 20% by weight and preferably about 3 to about 10% by weight.

Fluoride ion releasing salts are incorporated in the dentifrice composition of the present invention and are characterized by their ability to release fluoride ions in water. It is preferable to employ a water soluble fluoride salt providing about 1000 to about 9000 ppm of fluoride ion, and preferably about 2500 to about 8800 ppm of fluoride ion. Suitable examples of fluoride ion releasing salts include water soluble inorganic metal salts, for example, sodium fluoride, potassium fluoride, sodium monofluorophosphate, stannous fluoride and sodium fluorosilicate. Sodium fluoride, sodium monoflurophosphate and stannous fluoride are preferred fluoride ion releasing salts.

Pyrophosphate salts having anticalculus efficacy useful in the practice of the present invention include water soluble salts such as dialkali or tetraalkali metal pyrophosphate salts such as $Na_4P_2O_7$ (TSPP), $K_4P_2O_7$, $Na_2K_2P_2O_7$, $Na_2H_2P_2O_7$ and $K_2H_2P_2O_7$. Polyphosphate salts include the water soluble alkali metal tripolyphosphates such as sodium tripolyphosphate and potassium tripolyphosphate.

The pyrophosphate salts are incorporated in the dentifrice composition of the present invention at a concentration of about 0.5 to about 2.0% by weight, and preferably about 1.5 to about 2% by weight and the polyphosphate salts are incorporated in the dentifrice composition of the present invention at a concentration of about 1.0 to about 7.0% by weight.

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

Dyes used in the practice of the present invention are generally food color additives presently certified under the Food Drug & Cosmetic Act for use in the food and ingested drugs, including dyes such as FD&C Red No. 3 (sodium salt of tetraiodofluorescein), FD&C Yellow No. 5 (sodium salt of 4-p-sulfophenylazo-1-p-sulfophenyl-5-hydroxypyrazole-3 carboxylic acid), FD&C Yellow No. 6 (sodium salt of p-sulfophenylazo-B-naphtol-6-monosulfonate), FD&C Green No. 3 (disodium slat of 4-{[4-(N-ethyl-p-sulffobenzylamino)-phenyl]-(4-hydroxy-2-sulfoniumphenyl)-mewthylene}-[1-(N-ethyl-N-p-sulfobenzyl)-⊗-3,5-cyclohexadienimine], FD&C Blue No. 1 (disodium salt of dibenzyldiethyldiaminotriphenylcarbinol trisulfonic acid of indigotin) and mixtures thereof in various proportions. The concentration of the dye for the most effective result in the present invention is present in the dentifrice composition in an amount from about 0.0005 percent to about 2 percent of the total weight.

A striped dentifrice product may be obtained using the dual component dentifrice embodiment 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 the pigments and dyes discussed above.

Any suitable flavoring or sweetening material may also be incorporated in the dentifrice composition of the present invention. 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, xylitol, sodium cyclamate, perillatine, and sodium saccharin. Suitably, flavor and sweetening agents may together comprise from 0.01% to 5% or more of the preparations.

Antibacterial agents are non-cationic antibacterial agents based on phenolic and bisphenolic compounds, halogenated diphenyl ethers such as Triclosan, benzoate esters and carbanilides as well as cationic antibacterial agents such as chlorhexidine digluconate. Such antibacterial agents can be present in quantities of from about 0.03 to about 1% by weight of the particular component.

When noncationic antibacterial agents or antibacterial agents are included in any of the dentifrice components, there is also preferably included from about 0.05 to about 5% of an agent which enhances the delivery and retention of the agents to, and retention thereof on oral surfaces. Such agents useful in the present invention are disclosed in U.S. Patents 5,188,821 and 5,192,531; and include synthetic anionic polymeric polycarboxylates, such as 1:4 to 4:1 copolymers of maleic anhydride or acid with another polymerizable ethylenically unsaturated monomer, preferably methyl vinyl ether/maleic anhydride having a molecular weight (M.W.) of about 30,000 to about 1,000,000, most preferably about 30,000 to about 800,000. These copolymers are available for example as Gantrez. e.g. AN 139 (M.W. 500,000), AN 119 (M.W. 250,000) and preferably S-97 Pharmaceutical Grade (M.W. 700,000) available from ISP Technologies, Inc., Bound Brook, N.J. 08805. The enhancing agents when present are present in amounts ranging from 0.05 to about 3% by weight.

To prepare the dentifrice components of the present invention, generally the humectants, for example, propylene glycol, polyethylene glycol ingredients, are dispersed with any organic thickeners, sweetener, pigments such as titanium dioxide and any polyphosphates included as anti-calculus ingredients. Water is then added into this dispersion along with any antibacterial agent such as Triclosan, any antibacterial enhancing agent such as Gantrez and any anticalculus additional agents. In the first neutral pH component a fluoride ion source desensitizing agent and phosphate buffering agent is added. In the second component an ingredient to adjust the pH to an alkaline level is added, such as sodium hydroxide. These ingredients are mixed until a homogenous phase is obtained for each component. Thereafter inorganic thickener, silica abrasive, flavor and surfactant ingredients are added and the ingredients mixed at high speed under vacuum of from about 20 to 100 mm of Hg. The resultant product, in the case of each component, is a homogeneous, semi-solid, extrudible paste product.

The dentifrice composition may be applied to hypersensitive tooth surfaces in the form of a paste or gel by tooth brushing or topically applied by being painted directly on the tooth surfaces in the form of a liquid varnish using a soft applicator brush.

The single phase dentifrice composition embodiment of the present invention may be packaged in a single tube or other conventional package. The multicomponent dentifrice composition embodiment of the present invention is packaged in a suitable dispensing container in which the components are maintained physically separated and from which the separated components may be dispensed synchronously as a combined ribbon for application to a toothbrush. Such containers are known in the art. An example of such a container is a two compartment dispensing container, such as a pump or a tube, having collapsible sidewalls, as disclosed in U.S. Patents 4,487,757 and 4,687,663; wherein, the tube body is formed from a collapsible plastic web such as polyethylene or polypropylene and is provided with a partition within the container body defining separate compartments in which the physically separated components are stored and from which they are dispensed through a suitable dispensing outlet.

The following example is further illustrative of the present invention, but it is understood that the invention is not limited thereto. All amounts and proportions referred to herein and in the appended claims are by weight, unless otherwise stated.

Example I

A two component (Component A and B) desensitizing dentifrice of the present invention was prepared, designated "Dentifrice X", Component A having neutral pH (6.5) and a Component B having an alkaline pH (9.5). When combined in equal amounts for tooth brushing, Dentifrice X had a pH of 7.5 in a 1:3 slurry with water. The ingredients of Components A and B are listed in Table I below.

TABLE I

Component Ingredients	Dentifrice X Weight % A	B
Deionized Water	32.995	36.895
Sodium Fluoride	1.105*	1.105*
Potassium Nitrate	5.00	5.00
Glycerin	18.000	18.000
Polyethylene glycol 600	3.000	3.000
Xanthan gum	7.000	7.000
Carboxymethyl cellulose	0.500	0.500
Sorbitol 70% NC	5.00	5.000
Sodium saccharin	0.400	0.400
Titanium Dioxide	---	1.000
Pluronic F-127	2.000	2.000
Sodium Hydroxide (50%)	---	1.000
Sodium Phosphate Mono	4.000	---
Sodium Phosphate Dibasic	3.500	---
FD&C Blue #1 (1.25% solution)	---	0.300
Zeodent 115	20.000	15.000
Zeodent 165	1.000	1.500
Sodium bicarbonate	---	2.500
N-silicate	---	3.800
Sodium lauryl sulfate	1.500	1.500
Flavor	1.100	1.100

*Releases 5000 ppm fluoride ion

In the preparation of Dentifrice X, components A and B were prepared wherein the glycerin, polyethylene glycol and organic thickeners were dispersed in a conventional mixer until the mixture became a slurry, which was smooth in appearance. Color and sweetener were dispersed in this slurry before the addition of water. In the preparation of Component A, potassium nitrate was then dispersed in this slurry. In the preparation of Component B, sodium hydroxide was then dispensed in the gel phase. This mixture was mixed for 20 to 30 minutes producing a homogeneous gel phase. The mixture was added to a vacuum mixer and cooled below 105°F. Zeodent 115, Zeodent 165 and sodium bicarbonate were then added and mixed for 10 to 30 minutes at high speed under a vacuum of about 50 mm Hg, providing a homogenous mixture. The sodium lauryl sulfate and flavor were then added to the individual dentifrice components which was followed by mixing another 5-15 minutes under vacuum of 50 mm Hg to prepare the resultant component product.

The desensitizing efficacy of the two component Dentifrice X was evaluated using 4.25mm X 4.25mm square dentin disks of 750μm thickness cut from extracted human molars. The disks were prepared for treatment by etching with 6% citric acid for 2 minutes to remove any surface smear.

For purposes of comparison the procedure of the Example I was repeated with another group of similarly prepared disks using a dual component dentifrice designated "Dentifrice Y", comparable to that of US 6,180,089 in which the alkaline component designated "Component C" had a pH of 9.5 and the acidic component designated "Component D" had a pH of 5.2. The ingredients of Components C and D of Dentifrice Y are listed in Table II below.

As a control, the procedure of the Example was repeated using a phosphate buffer solution as the treatment which treatment was designated "Control". The ingredients of the phosphate buffer solution are listed in Table III below.

The ingredients of Components C and D of Dentifrice Y are listed in Table II below.

TABLE II

Dentifrice Y

Component C		Component D	
Ingredient	**%**	**Ingredient**	**%**
Deionized water	29.57	Dionized water	25.66
Potassium nitrate	10.000	Anhydrous citric acid	0.531
Glycerin	25.48	Sodium citrate	2.657
PEG 600	3.00	Stannous chloride	0.600
Xanthan NF	0.700	Stannous fluoride	0.908*
Sodium carboxymethyl cellulose	0.50	Glycerin	33.704
Sodium saccharin	0.4	Xanthan	0.500
Titanium dioxide	2.00	Sodium carboxymethyl cellulose 2000S	0.700
Pluoronic F-127	1.00	Sodium saccharin	0.400
Zeodent 115	15.00	Tetrasodium pyrophosphate	0.500
Zeodent 165	1.75	FD&C Blue #1 (1.25% soln.)	0.240
Sodium bicarbonate	5.00	PEG 40 oil	6.00
Sodium hydroxide	3.00	Pluoronic F-127	2.00
Flavor stannous plus	1.10	Zeodent 115	20.00
Sodium lauryl sulfate	1.5	Zeodent 165	3.00
		Flavor	1.100
		Sodium lauryl sulfate	1.500
Totals	**100**		**100**

*Releases 2200 ppm fluoride ion. Fluoride ion delivery of Dentifrice Y is 1100 ppm when Component C and D are combined for use.

TABLE III

Phosphate Buffer Solution

Ingredient	Wt. %	Millimoles
Sodium phosphate mono	0.0087	0.63
$CaCl_2$	1.1456	1.06
NaCl	0.877	150.0

The etched disks were then treated by separately brushing the discs for a 60 second period with either Dentifrice X or Y or the phosphate buffer solution (control).

The surface composition of the treated disks were then subjected to Electron Spectroscopy for Chemical Analysis (ESCA) and Scanning Electron Microscopy (SEM) analysis. The ESCA results are recorded in Table IV below as an average for each group. The percentage of nitrogen on the dentin surface is generally attributed to the amount of exposed collagen material which is an integral part of the dentin structure. The reduced amount of nitrogen is indicative of a surface coating, and the higher the amount of calcium ion, the greater the degree of tubular occultation.

TABLE IV

ESCA Analysis

Atomic Percent

Dentifrice	C	O	N	Ca	P	Si	Na	Sn	F	P/Ca Ration
X	33.20	42.63	3.78	7.66	6.32	4.88	0.80	0.19	0.55	0.82
Y	27.76	47.27	2.45	4.73	4.10	11.39	1.02	1.16	0.11	0.86
Control	59.69	22.78	14.72	1.17	0.99	0.68	--	--	--	0.85

The results recorded in Table IV indicate that the amount of deposit formed on the surface of the dentin disks treated with the combined components of Dentifrice X of the present invention is substantially greater than the disks treated with comparative Dentifrice Y, indicating a significantly greater degree of tubular obturation would be experienced with the use of Dentifrice X as compared to Dentifrice Y.

The SEM photomicrographs taken of the dentin surfaces subjected to the brushing treatments are shown in Figures 1-3 respectively. Examination of the SEM of the Dentifrice X treated dentin disk surface, (Figure 1), indicates that dentinal tubule obturation was substantially complete as compared to treatment with comparative dual component Dentifrice Y as indicated by examination of the photomicrograph of Fig. 2. The Control treatment of the dentin disks using an phosphate buffer solution as shown in the SEM of Fig. 3 indicates a limited amount of dentinal tubule obturation.

The ESCA and the SEM results all provide evidence that the unique combination of the neutral and alkaline potassium ion containing dentifrice components in combination with high releasable fluoride ion concentration levels effects an unexpected significant improvement in the remediation of dentinal hypersensitivity.

Example II

A single component dentifrice designated Dentifrice Z was prepared which contained the ingredients listed in Table V below. The dentifrice composition has a pH of 8.5.

TABLE V

Ingredients	Dentifrice Z (Wt.%)
Deionized Water	34.345
Sodium Fluoride	1.105*
Potassium Nitrate	5.00
Glycerin	18.000
Polyethylene glycol 600	3.000
Xanthan gum	0.7000
Carboxymethyl cellulose	0.500
Sorbitol 70% NC	5.00
Sodium saccharin	0.400
Pluronic F-127	2.000
Sodium phosphate mono	2.0
Sodium phosphate diabasic	1.75
FD&C Blue #1 (1.25% solution)	0.2
Zeodent 115	17.5
Zeodent 165	1.25000
Sodium Bicarbonate	1.75
Sodium lauryl sulfate	1.500
Flavor	1.100

*Releases 5000 ppm fluoride ion

In-Vitro Remineralization/Demineralization Test with Fluoride Uptake

- **Specimen Preparation**

An in vitro fluoride uptake test method using enamel specimens (3 mm diameter) removed from extracted human teeth and mounted in rods to evaluate the performance of Dentifrice 2. The specimens were ground and polished to a high luster with Gamma Alumina using standard methods. Eighteen specimens per group were prepared for this study.

- **Initial Decalcification**

Artificial lesions were formed in the enamel specimens by a 96-hour immersion into a solution of 0.1 M lactic acid and 0.2% Carbopol C907 which was 50% saturated with hydroxyapatite and adjusted to pH 5.0. The lesion surface hardness range was 25-45 and average lesion depth was approximately 70μ.

- **Saliva Collection**

A 50:50 mixture of pooled, human saliva and a mineral solution was used as the remineralization medium in all treatment regimens. Wax-stimulated saliva was collected from at least five individuals and refrigerated until used. Saliva samples were then pooled with vigorous stirring prior to distribution (7.5 ml + 7.5 ml mineral mix) into 30 ml treatment beakers. Fresh saliva/mineral mix was used each day (changed during the acid challenge period).

- **Treatment Slurries**

During the treatment period, the specimens were immersed in dentifrice slurries to simulate daily brushing. The slurries were prepared by adding 5.0 g of dentifrice to 10 g of the fresh saliva/mineral mix solution in a beaker with a magnetic stirrer. A fresh slurry was prepared just prior to each treatment.

- **Treatment Regimen**

The cyclic treatment regimen consisted of a 4.0 hour/day acid challenge in the lesion forming solution described above with four, one-minute dentifrice treatment periods. After the treatments, the specimens were rinsed with running distilled water and then replaced back into the saliva. The remaining time (about 20 hours) the specimens were in the pooled, human saliva, remineralization system. The regimen was repeated for 20 days. The treatment schedule used was as follows:

a.	8:00-8:01 a.m.	Dentifrice treatment*
b.	8:01-9:00 a.m.	Saliva treatment
c.	9:00-9:01 a.m.	Dentifrice treatment
d.	9:01-10:00 a.m.	Saliva treatment
e.	10:00 a.m.-2:00 p.m.	Acid challenge
f.	2:00-3:00 p.m.	Saliva treatment
g.	3:00-3:01 p.m.	Dentifrice treatment
h.	3:01-4:00 p.m.	Saliva treatment
i.	4:00-4:01 p.m.	Dentifrice treatment
j.	4:01 p.m.-8:00 a.m.	Saliva treatment
k.	Back to (a)	

* On the first day, this treatment was not given; the test began with one hour in saliva to permit pellicle development prior to any treatments.

- **Fluoride Analysis**

At the end of the 20-day treatment regimen, the fluoride content of each enamel specimen was determined using the microdrill technique to a depth of 100μ. Fluoride data were calculated as μg F/cm^3: (μg F X dilution factor – volume of drilling). The results of the fluoride analysis are recorded in Table VI below.

For purposes of comparison, a phase dentifrice designated "Composition C", which had a fluoride ion content of 5000 ppm in which potassium nitrate was absent was also tested for fluoride ion uptake. Composition C had a pH of 7.5. The fluoride uptake for Composition C is also recorded in Table VI.

Table VI

Fluoride Uptake Assay Results

Dentifrice	Fluoride ion Uptake ug/cm^3)	Standard Deviation
Z	8128	±131
C	1987	±341

The fluoride uptake data recorded in Table VI indicate that the single phase composition that contained 5% potassium nitrate in which the pH was 9.0 unexpectedly deposited substantially more fluoride ion about four times as much than a comparative single phase composition in which the pH was 8.0 and potassium nitrate was not present in the dentifrice.

Dentifrice Z was tested in vitro for in situ remineralization of caries lesions in employing the methodology described below.

- **Remineralization Measurements**

  Following fluoride analysis discussed above all samples were tested for surface hardness changes. The difference between the hardness following treatment and initial lesion hardness indicated the ability of that treatment to enhance remineralization.

- **Determination of Enamel Resistance to Demineralization**

  The resistance of the treated enamel to a subsequent acid challenge was determined by placing the treated specimens into the lesion formation solution (with no remineralization phase) for one 2-hour and one 16-hour period of simulated plaque acid challenge (SPAC). Following each acid challenge, the surface hardness of the specimens was measured as a Vickers Hardness Number (VHN) using a Leite Microhardness tester. The difference between the hardness following each subsequent demineralization and the initial lesion hardness reflected the degree of resistance to demineralization provided by each dentifrice.

Results of the remineralization tests of Dentifrice Z are recorded in Table VII below.

For purposes of comparison a toothpaste product designated Dentifrice D having a composition substantially similar to Dentifrice Z except that potassium nitrate was not included in Dentifrice D was also tested for remineralization efficacy. The pH of Dentifrice D was 7.5. The remineralization results for Dentifrice D are also recorded in Table VII below.

Table VII
Enamel Hardness

Composition	ΔVHN
Z	133±8
D	21±4

The desensitizing efficacy of Dentifrice Z was evaluated following the procedure of Example I.

For purposes of comparison the procedure of the Example I was repeated with another group of similarly prepared disks using a dentifrice designated "Dentifrice E". Dentifrice E differed from Dentifrice Z in that the fluoride content was 1100 ppm Fluoride and did not contain phosphate buffering salts.

Electron Spectroscopy for Chemical Analysis (ESCA) and Scanning Electron Microscopy (SEM) analysis results are recorded in Table VIII below.

TABLE VIII

ESCA ANALYSIS

	Atomic Percent									Ratio
Dentifrice	**C**	**O**	**N**	**Ca**	**P**	**Si**	**Na**	**Sn**	**F**	**P/Ca**
E	33.05	42.46	5.75	3.81	3.14	10.19	0.70	0.82	0.09	0.83
Z	32.06	43.37	1.14	10.78	8.68	2.10	0.96	--	0.93	0.81

The results recorded in Table VIII indicate that the amount of Ca and deposited on the surface of the dentin disks treated with Dentifrice Z of the present invention was significantly greater, that is, three times greater than the disks treated with comparative Dentifrice E, indicating a significantly greater degree of tubular obturation would be experienced with the use of Dentifrice Z as compared to Dentifrice E.

CLAIMS

What is claimed is:

1. A dental composition which eliminates or substantially reduces the discomfort and pain associated with dentinal hypersensitivity and exhibits enhanced anticaries and remineralization benefits which composition comprises a fluoride ion and a potassium ion releasable salt, the composition having a pH in the range of about 7.5 to about 9, the pH being buffered with an alkali metal phosphate salt, whereby application to teeth requiring relief from dentine hypersensitivity results in heightened desensitization as well as heightened tooth fluoridation and remineralization.

2. The composition of claim 1 wherein the potassium ion releasable salt is potassium nitrate.

3. The composition of claim 1 wherein the fluoride releasable salt present in the composition delivers a fluoride ion concentration of about 1100 to about 8800 ppm.

4. The composition of claim 1 wherein the fluoride ion releasable salt is sodium fluoride.

5. The composition of claim 1 wherein the pH is buffered with a sodium phosphate salt.

6. The composition of claim 1 wherein the pH is buffered with a combination of sodium mono and di- basic phosphate salts.

7. The composition of claim 1 wherein the alkali metal phosphate salt is present in the composition at a concentration of about 1.0 to about 10% by weight.

8. A method for eliminating or reducing the discomfort and pain associated with dentinal hypersensitivity concomitant with enhancing tooth remineralization and reducing the occurrence of caries, which method comprises preparing a dentifrice having a pH buffered with a phosphate salt in the range of about 7.5 to about 9.0 and containing a desensitizing potassium ion releasable salt and a fluoride ion releasable salt, dispensing the composition for application to teeth requiring relief from dentine hypersensitivity and thereafter applying the composition to the teeth whereby heightened tooth desensitization, enhanced tooth remineralization and increased fluoride uptake is experienced by the user.

9. The method of claim 6 wherein the potassium ion releasable salt is potassium nitrate.

10. The method of claim 8 wherein the fluoride salt present in the composition delivers a fluoride ion concentration of about 1100 to about 8800 ppm.

11. The method of claim 8 wherein the fluoride ion releasable salt is sodium fluoride.

12. The method of claim 8 wherein the alkaline dentifrice component is an aqueous dentifrice having a pH of about 9 to about 11.

13. A two component dental composition which eliminates or substantially reduces the discomfort and pain associated with dentinal hypersensitivity which composition comprises a first dentifrice component having a neutral pH in the range of about 6.5 to 7.5, the pH being buffered with a phosphate salt, a second dentifrice component having an alkaline pH in the range of about 8.0 to about 9.9, and at least one of the components containing a fluoride ion or potassium ion releasable salt, the first and second components being maintained separate from each other until dispensed and combined for application to teeth requiring relief from dentine hypersensitivity, whereby heightened desensitization enhanced remineralization and increased fluoride uptake is experienced by the user.

14. The composition of claim 13 wherein each component contains a fluoride ion and a potassium ion releasable salt.

15. The composition of claim 13 wherein the potassium ion releasable salt is potassium nitrate.

16. The composition of claim 13 wherein the fluoride salt present in the dual component composition delivers a fluoride ion concentration of about 2500 to about 8800 ppm.

17. The composition of claim 13 wherein the fluoride ion releasable salt is sodium fluoride.

18. The composition of claim 13 wherein the alkaline dentifrice component is an aqueous dentifrice having a pH of about 8.0 to about 9.9..

19. The method of claim 13 wherein the pH of the neutral pH dentifrice component is buffered with a sodium phosphate salt.

20. The composition of claim 13 wherein the pH of the alkaline dentifrice component is adjusted with sodium hydroxide.

21. The method for eliminating or reducing the discomfort and pain associated with dentinal hypersensitivity which comprises preparing (1) a first dentifrice component having a neutral pH buffered with a phosphate salt in the range of about 6.5 to about 7.5 and (2) a second dentifrice component having an alkaline pH in the range of about 8.0 to about 9.9, at least one of the components containing a desensitizing potassium ion releasable salt and a fluoride ion releasable salt, separately housing the first and second components, dispensing the first and second components and combining the dispensed components for application to teeth requiring relief from dentine hypersensitivity and thereafter applying the combined components to the teeth whereby heightened desensitization is experienced by the user.

22. The method of claim 21 wherein each component contains a fluoride ion and a potassium ion releasable salt.

23. The method of claim 21 wherein the potassium ion releasable salt is potassium nitrate.

24. The method of claim 21 wherein the fluoride salt present in the composition delivers a fluoride ion concentration of about 1100 to about 8800 ppm.

25. The method of claim 21 wherein the fluoride ion releasable salt is sodium fluoride.

26. The method of claim 21 wherein the alkaline dentifrice component is an aqueous dentifrice having a pH of about 8.0 to about 9.9.

27. The method of claim 21 wherein the pH of the alkaline dentifrice component is adjusted with sodium hydroxide.

28. The method of claim 21 wherein the pH of the neutral pH dentifrice is buffered with a sodium phosphate salt.

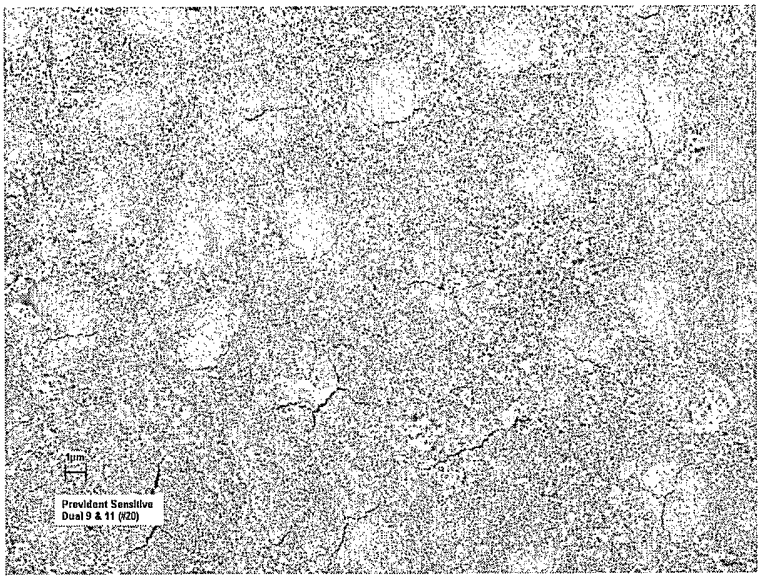

Figure 1

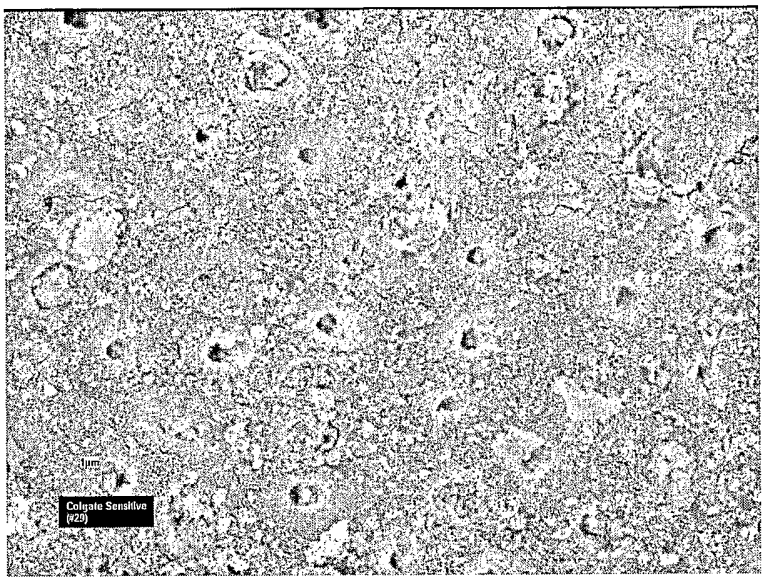

Figure 2

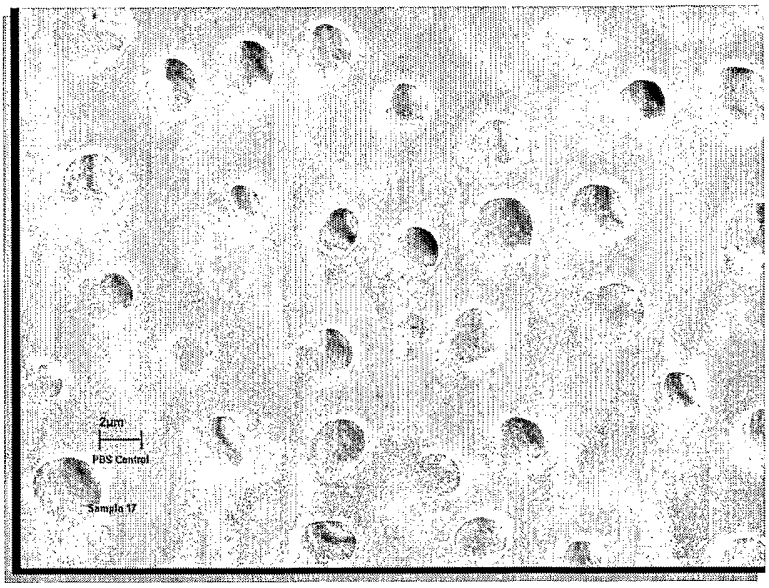

Figure 3