



(51) International Patent Classification:

A61K 8/19 (2006.01) *A61K 8/34* (2006.01)
A61K 8/26 (2006.01) *A61Q 11/00* (2006.01)

(21) International Application Number:

PCT/EP2014/055891

(22) International Filing Date:

25 March 2014 (25.03.2014)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

1399/MUM/2013 15 April 2013 (15.04.2013) IN
13169725.2 29 May 2013 (29.05.2013) EP

(71) **Applicant** (for all designated States except AE, AG, AU, BB, BH, BN, BW, BZ, CA, CY, EG, GB, GD, GH, GM, IE, IL, IN, KE, KN, LC, LK, LS, MT, MW, MY, NA, NG, NZ, OM, PG, QA, RW, SA, SC, SD, SG, SL, SZ, TT, TZ, UG, US, VC, ZA, ZM, ZW): **UNILEVER N.V.** [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL).

(71) **Applicant** (for AE, AG, AU, BB, BH, BN, BW, BZ, CA, CY, EG, GB, GD, GH, GM, IE, IL, IN, KE, KN, LC, LK, LS, MT, MW, MY, NA, NG, NZ, OM, PG, QA, RW, SA, SC, SD, SG, SL, SZ, TT, TZ, UG, VC, ZA, ZM, ZW only): **UNILEVER PLC** [GB/GB]; a company registered in England and Wales under company no. 41424 of Unilever House, 100 Victoria Embankment, London Greater London EC4Y 0DY (GB).

(71) **Applicant** (for US only): **CONOPCO, INC., D/B/A UNILEVER** [US/US]; 800 Sylvan Avenue, AG West, S. Wing, Englewood Cliffs, New Jersey 07632 (US).

(72) **Inventors**: **CHANDRASEKARAN, Sembian**; Hindustan Unilever Ltd, Unilever House, BD Sawant Marg, Chakala, Andheri (East), Mumbai 400 099 (IN). **IYER, Meenakshi**; Hindustan Unilever Ltd, Unilever House, BD Sawant Marg, Chakala, Andheri (East), Mumbai 400 099 (IN). **PATIL, Subhash, Madhav**; Hindustan Unilever Ltd, Research Centre, B.D. Sawant Marg, Chakala, Andheri (East), Mumbai 560 035 (IN).

(74) **Agent**: **TANSLEY, Sally, E**; Unilever Patent Group, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL).

(81) **Designated States** (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) **Designated States** (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:

- as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))
- as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))
- of inventorship (Rule 4.17(iv))

Published:

- with international search report (Art. 21(3))
- before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))

(54) **Title**: TOOTHPASTE CONTAINING SMECTITE CLAY

(57) **Abstract**: Disclosed is a toothpaste composition comprising: (i) 20 to 60 wt% of a calcium based abrasive; (ii) 3 to 15 wt% humectant; and, (iii) upto 2 wt% thickening silica; wherein the composition comprises 0.2 to 3 wt% smectite clay. Disclosed composition provides superior sensory profile as well as an increased uptake of antibacterial agent by the oral tissue.



WO 2014/170096 A1

TOOTHPASTE CONTAINING SMECTITE CLAY

Field of the Invention

The present invention relates to toothpaste composition having superior sensory profile and antibacterial action.

Background of the invention

Toothpaste is a popular dentifrice product which contains an abrasive, one or more humectants, antibacterial agent, a surfactant, thickening or binding agents and flavor.

Variety of antibacterial agents is used in oral care products. For example, halogenated hydroxydiphenyl ether compounds such as Triclosan[®] (TCN) have been used in oral care compositions to counter plaque. Effect of an antibacterial agent is largely dependent on delivery and uptake inside the oral cavity.

US6451291B1 (Colgate, 2002) discloses that mixed anionic/zwitterionic lead to an increase in the uptake of antibacterial agent which enhances the therapeutic efficacy of the agent.

US5188821A (Colgate, 1993) discloses that an anionic polymeric polycarboxylate, for example, Gantrez[®] of GAF Corporation, is a good antibacterial enhancing agent.

In US2009269287A (P&G), is disclosed an oral care composition where traditional humectant components such as glycerin, sorbitol and other polyols are, to some extent, replaced by a binding/thickening system having select carrageenans. The dentifrice compositions exhibit increased in-use dispersibility in saliva which provides for an increased contact time of the composition with the user's teeth and oral cavity tissues and therefore the active agents are more rapidly available for beneficial activity.

However there is still an unmet need to enhance delivery and uptake of antibacterial agents.

In an attempt to provide cost-effective toothpaste compositions, the present inventors tried to lower the humectants level. However, as expected, drastic reduction in humectants led to products with poorer sensory properties which may not find consumer acceptance. This posed a technical problem of how to balance the sensory properties whilst still being able to minimize costs.

Surprisingly, it has been determined that reduction in dosage of thickening silica and introduction of smectite clay provides a solution.

Summary of the Invention

In accordance with a first aspect is disclosed a toothpaste composition having:

- (i) 20 to 60 wt% of a calcium based abrasive;
- (ii) 3 to 15 wt% humectant; and,
- (iii) upto 2 wt% thickening silica;

wherein said composition comprises 0.2 to 3 wt% smectite clay.

In accordance with a second aspect is disclosed a method of promoting oral hygiene comprising a step of applying to the teeth an effective amount of a toothpaste composition of the first aspect.

In accordance with a third aspect is disclosed a toothpaste composition of the first aspect for promoting improved oral hygiene.

For better understanding of the invention, reference should be made to the following detailed description of preferred embodiments.

Detailed Description of the Invention

In accordance with a first aspect is disclosed a toothpaste composition having:

- (i) 20 to 60 wt% of a calcium based abrasive;
- (ii) 3 to 15 wt% humectant; and,
- (iii) upto 2 wt% thickening silica;

wherein the composition includes 0.2 to 3 wt% smectite clay.

Calcium containing abrasive

All toothpastes generally contain an abrasive agent. Gels usually contain silica, whereas creams contain calcium based abrasives, especially calcium carbonate. The quantity of abrasives needs to be balanced otherwise there may be excessive abrasion.

Preferred toothpaste compositions have 25 to 60 wt% of calcium based abrasive, more preferably 30 to 55 wt% and most preferably 40 to 55 wt% of calcium containing abrasive.

A particularly preferred abrasive is calcium carbonate. Fine ground natural chalk (FGNC), a form of chalk, is highly preferred.

FGNC may also be modified chemically or physically by coating during milling or after milling by heat treatment. Typical coating materials include magnesium stearate and oleate. The morphology of FGNC may also be modified during the milling process by using different milling techniques, for example, ball milling, air-classifier milling or spiral jet milling.

FGNC can be used as the sole calcium based abrasive. However, FGNC can also be used with other calcium based abrasives to balance the abrasion. Usually the particle size of chalk is from 1 to 60 μm , and the preferred size range from 1 to 15 μm .

Other preferred calcium based abrasives include dicalcium phosphate (DCP), calcium pyrophosphate and precipitated calcium carbonate (PCC).

When a combination of Calcium based abrasives is used, it is preferred that FGNC is 35 to 100%, more preferably 75 to 100% and especially from 95 to 100% of the whole of the Calcium based abrasives. In such cases, the balance, most preferably, is PCC (precipitated calcium carbonate).

Other abrasives may also be used along with the calcium based abrasive, depending upon the intended degree of abrasion. These include synthetic abrasive polishing agents such as amorphous precipitated silica and silica gels. Other abrasive agents include magnesium carbonate, sodium metaphosphate, potassium metaphosphate, zirconium silicate, potassium metaphosphate, magnesium orthophosphate, tricalcium phosphate, magnesium orthophosphate, trimagnesium phosphate, aluminum silicate, zirconium silicate and perlite.

Thickening silica

The disclosed composition contains upto 2 wt% thickening silica. Conventional gels contain upto 8 wt% thickening silica whereas opaque products typically have 4 to 10 wt%. Excessive thickening silica causes an adverse effect on release of flavor ingredients. Preferred compositions have 1.5 wt%, or even less than 1 wt% thickening silica. Optimal compositions have less than 0.5 wt% thickening silica.

Preferred thickening silicas include AEROSIL® T 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. Useful silica thickeners also include ZEODENT® 165, ZEODENT® 163 and/or 167 and ZEOFREE® 153, 177, and/or 265 silicas, all available from J. M. Huber Corporation. Other preferred thickening silicas include MFIL®, MFIL®-P (From Madhu Silica), SIDENT® 22 S and AEROSIL® 200 (Ex. Evonik Industries), SYLODENT® and PERKASIL® thickening silicas from WR Grace & Company and Tixosil® 43 and 331 from Rhodia, synthetic finely divided pyrogenic silica such as those sold under the trademarks SYLOID® 244, SYLOID® 266 and AEROSIL® D-200.

Smectite clay

Toothpaste compositions typically contain about 20 wt% humectants, which is usually a combination of humectants. This necessitates the inclusion of from about 4 to 6 wt% thickening silica. Such toothpastes have excellent sensory profile, therefore highly preferred by consumers. On the other hand, products with humectants of about 6 to 10 wt% have less preferred sensory profile which may get further affected on reducing the silica content.

A technical solution is provided by smectite clay. Smectites constitute a group in the class of natural aluminosilicate minerals known as layered silicates.

A preferred smectite clay is selected from phyllosilicates, montmorillonites, bentonites, hectorites and derivatives thereof, purified magnesium aluminium silicates, purified sodium magnesium silicates organically modified smectite clay, organically modified montmorillonite clay and mixtures thereof.

Further preferred smectite clay is selected from montmorillonites (bentonites, hectorites and derivatives thereof); purified magnesium aluminium silicates (various grades are commercially available as VEEGUM® from R. T. Vanderbilt Company); purified sodium magnesium silicates (commercially available as LAPONITE® in various grades); organically modified smectites including tetra alkyl and/or trialkyl ammonium smectites (organically modified montmorillonite clays) such as quaternium-18 bentonite, quaternium-18 hectorite, stearylalkonium bentonite and stearylalkonium hectorite/ and mixtures thereof. Magnesium aluminium silicate clays are particularly preferred. An example is VEEGUM® HV. The clay tends to swell when exposed to water.

Preferred toothpaste compositions contain 0.2 to 3 wt% clay. More preferred compositions have 0.5 to 1 wt% clay.

The smectite clay is believed to not only play a role in aiding the sensory profile, but also in thickening the composition.

In preferred toothpastes, ratio of the calcium containing abrasive to the smectite clay is in the range of 1:0.1 to 1: 0.003.

Humectants

Disclosed compositions have 3 to 15 wt%, more preferably 4 to 12 wt% and most preferably 6 to 12 wt% total humectant. A particularly preferred humectant is sorbitol which is generally available in the form of 70% aqueous solution. Therefore, where sorbitol is used as a humectant, its actual dosage in the formulation is higher than the desired percentage to account for the water in the commercial product.

Other preferred humectants include glycerine, maltitol and xylitol. More preferred toothpastes contain glycerine and sorbitol for lubricated mouth feel, but their cumulative levels should not exceed the disclosed upper limits. Lower humectant content also provides an effective way to reduce the cost of the product.

Antibacterial agent

Preferred toothpaste compositions include 0.05 to 5 wt% of the antibacterial agent.

Suitable examples include quaternary ammonium compounds such as cetylpyridinium chloride; bis-biguanides such as chlorhexidine, chlorhexidine digluconate, hexetidine, octenidine, alexidine, TRICLOSAN[®] and other halogenated bisphenolic compounds such as 2,2' methylenebis-(4-chloro-6-bromophenol). A particularly preferred antibacterial agent is TRICLOSAN[®]; a broad-spectrum antimicrobial agent widely used in oral care products. In compositions which include an antibacterial agent, preferably triclosan, we have observed significantly high uptake of the antibacterial agent by dental tissues including teeth. This is a hitherto unreported technical effect which is believed to be due to the presence of smectite clay.

Other binders

Preferred toothpaste compositions may also have one or more other binder. Cellulosic binders are especially preferred. Preferred cellulosic binders include cellulose ethers, which include hydroxyethyl cellulose (HEC), hydroxypropyl cellulose (HPC), ethylhydroxyethyl cellulose (EHEC), carboxymethyl cellulose (CMC), carboxymethylhydroxyethyl cellulose (CMHEC), hydroxypropylhydroxyethyl cellulose (HPHEC), methyl cellulose (MC), methylhydroxypropyl cellulose (MHPC),

methylhydroxyethyl cellulose (MHEC), carboxymethylmethyl cellulose (CMMC), hydrophobically modified carboxymethyl cellulose (HMCMC), hydrophobically modified hydroxyethyl cellulose (HMHEC), hydrophobically modified hydroxypropyl cellulose (HMHPC), hydrophobically modified ethylhydroxyethyl cellulose (HMEHEC), hydrophobically modified carboxymethylhydroxyethyl cellulose (HMCMHEC), hydrophobically modified hydroxypropylhydroxyethyl cellulose (HMHPHEC), hydrophobically modified methyl cellulose (HMMC), hydrophobically modified methylhydroxypropyl cellulose (HMMHPC), hydrophobically modified methylhydroxyethyl cellulose (HMMHEC), and hydrophobically modified carboxymethylmethyl cellulose (HMCMMC).

Other cellulosic binders include cationic hydroxyethyl cellulose (cationic HEC), cationic hydrophobically modified hydroxyethyl cellulose (cationic HMHEC) and microcrystalline cellulose.

A highly preferred binder is Sodium carboxymethyl cellulose (SCMC). Particularly preferred sodium carboxymethyl celluloses include those with degree of substitution of from 0.6 to 0.99, preferably from 0.7 to 0.95. Further, preferred SCMCs include those with viscosity of 250 mPa.s to 10000 mPa.s as measured on a Brookfield viscometer TA spindle at 30 rpm, 23 °C and reading after 30 seconds when slurried with flavour in a 1:1 ratio.

In addition to, or as a replacement of cellulosic binder, guar gum or its derivatives could be used. However, such binders are less preferred to the cellulosic binders. Such derivatives include carboxymethyl guar (CM guar), hydroxyethyl guar (HE guar), hydroxypropyl guar (HP guar), carboxymethylhydroxypropyl guar (CMHP guar), cationic guar, hydrophobically modified guar (HM guar), hydrophobically modified carboxymethyl guar (HMCM guar), hydrophobically modified hydroxyethyl guar (HMHE guar), hydrophobically modified hydroxypropyl guar (HMHP guar), cationic hydrophobically modified hydroxypropyl guar (cationic HMHP guar), hydrophobically modified carboxymethylhydroxypropyl guar (HMCMP guar) and hydrophobically modified cationic guar (HM cationic guar).

Other less preferred gums include xanthan gum, carrageenan and derivatives, such as Irish moss and viscarin, gellan gum, sclerotium gum and derivatives, pullulan, rhamosan gum, welan gum, konjac, curdlan, algin, alginic acid, alginates and derivatives, starch phosphate derivatives, agar and derivatives, gum arabic and derivatives, pectin and derivatives, chitosan and derivatives, karaya gum, locust bean gum, natto gum, tragacanth gum, chitin derivatives, gelatin, betaglucan, dextrin, dextran, cyclodextrin and polyquaterniums, furcellaren gum, ghatti gum, psyllium gum, quince gum, tamarind gum, larch gum, and tara gum.

Binders provide body to the compositions. In order to balance the viscosity and the binding ability of the composition, a balance needs to be struck between the smectite clay and the binder whenever it is present. For example, if more smectite clay is introduced into the composition, it may be preferable to lower the binder content. In the case of formulations having thickening silica and smectite clay and binder, their individual effects need to be taken into consideration because these actions usually produce a cumulative effect. For example, in the case of compositions containing thickening silica, the binder content needs careful control otherwise a viscous paste could result.

In the case of compositions which have cellulosic binder in addition to the smectite clay, the ratio of the amount of smectite clay to the cellulosic binder is in the range of 1:0.2 to 1:0.9. Excessive cellulosic binder leads to compositions which may show tailing effect while dispensing the composition on a toothbrush. On the other hand, most of the cellulosic binders and some others like carrageenan are expensive ingredients. Therefore, they use can improve the viscosity of the product, the option may not necessarily be commercially viable.

Other ingredients

In addition to the ingredients described earlier, preferred compositions may also include one or more other ingredients as described hereinafter.

Other thickening agents

Preferred toothpaste compositions may also include one or more other thickening agents such as carboxyvinyl polymers which include carbomers which are commercially available from B. F. Goodrich as the CARBOPOL® series, including CARBOPOL® 934, 940, 941 and 956.

Other preferred grades include acrylates/ C_{10-30} alkyl acrylate crosspolymers which are commercially available as ULTREZ® 21, PEMULEN® TR-1, and PEMULEN® TR-2, from Noveon Corporation. Preferred compositions can include 0.05 to 10 wt%, more preferably 0.1 to 5 wt%, and even more preferably 0.25 to about 4 wt% of other thickening agents.

De-sensitising agents

A de-sensitising agent is a potassium salt selected from potassium nitrate, potassium chloride, potassium citrate, potassium tartrate and potassium acetate used preferably from 0.5 to 3 wt%, more preferably from 1 to 2.5 wt % and especially from 1.7 to 2.2 wt%.

Silicate

Preferred toothpaste compositions can include alkali metal silicate. The alkali metal is sodium or potassium, preferably sodium. Sodium silicate is generally available as 10 to 40 % aqueous solution, most common being 30 % solution. Sodium silicate is available as neutral sodium silicate or alkaline sodium silicate. Preferred toothpastes have neutral sodium silicate. Sodium silicate is available with varying ratios of $Na_2O: SiO_2$.

Sodium silicate with $Na_2O: SiO_2$ ratio in the range of 3.0 to 3.8 is preferred, more highly preferred range being 3.25 to 3.5. Preferred toothpastes include 0.1 to 5 wt% silicate (on dry weight basis). Thus, a 30 % solution of sodium silicate is added to the composition in an amount in the range of 0.3 to 16 wt%.

Anti-caries agent

Preferred compositions can include one or more anti-caries agent. Such agents are typically fluorides. It is preferred that the source of fluoride is an alkali-metal salt of monofluorophosphoric acid, preferably sodium monofluorophosphate (SMFP).

SMFP is the fluoride source of choice when it comes to toothpaste compositions having Calcium based abrasives, especially chalk; since the alternative, sodium fluoride, reacts with the calcium carbonate to form insoluble calcium fluoride which has limited anti-caries activity. Preferred compositions include 0.01 to 2 wt%, more preferably 0.15 to 1 wt% and especially preferably 0.2 to 0.5 wt% anti-caries agent. It is preferable to maintain the free fluoride ion concentration from 100 to 2000 ppm, preferably from 900 to 1500 ppm. Other preferred anti-caries agents include sodium- and stannous fluoride, aminefluorides, sodium trimetaphosphate and casein.

Preservatives

Toothpastes with calcium containing abrasives, especially chalk; are prone to bacterial growth. Certain preservatives, e.g. methyl, ethyl, butyl, propyl and isopropyl esters of parahydroxybenzoic acid may be particularly useful against bacterial growth. A mixture of methyl, ethyl, butyl and propyl esters of parahydroxybenzoic acid is particularly preferred. The activity of this mixture can be enhanced by adding phenoxyethanol. Formaldehyde and dimethyl hydantoin are other preferred preservatives. Preservatives are generally included from 0.05 to 0.8 wt%.

Antioxidants

Preferred antioxidants are those which are compatible with other components and are not hazardous to health. These include ascorbic acid, ascorbyl palmitate, thiodipropionic acid, calcium ascorbate, dilauryldithiopropionate, gum guaiac, sodium ascorbate, butylated hydroxyl toluene, butylated hydroxyl anisole, and tocopherols. A mixture of antioxidants can be used.

When present, the antioxidant is added at a level effective to reduce or mitigate discoloration which would otherwise result from oxidation of the components of the toothpastes. Preferred levels are from 0.01 to 1 wt%.

Surfactants

Toothpastes generally contain surfactants, also commonly referred to as sudsing agents. Suitable surfactants are those which are reasonably stable and provide foam throughout a wide pH range. The surfactant may be anionic, nonionic, amphoteric, zwitterionic, cationic, or mixtures thereof.

Anionic surfactants useful herein include the water-soluble salts of alkyl sulfates having from 8 to 20 carbon atoms in the alkyl radical (e.g., sodium alkyl sulfate) and the water-soluble salts of sulfonated monoglycerides of fatty acids having from 8 to 20 carbon atoms. Sodium lauryl sulfate and sodium coconut monoglyceride sulfonates are examples of anionic surfactants of this type. Other suitable anionic surfactants are sarcosinates, such as sodium lauroyl sarcosinate, taurates, sodium lauryl sulfoacetate, sodium lauroyl isethionate, sodium laureth carboxylate, and sodium dodecyl benzenesulfonate.

Nonionic surfactants can be broadly defined as compounds produced by the condensation of alkylene oxide groups (hydrophilic in nature) with an organic hydrophobic compound which may be aliphatic or alkyl- aromatic in nature.

Examples of suitable nonionic surfactants include poloxamers (sold under trade name PLURONIC®), polyoxyethylene, polyoxyethylene sorbitan esters (sold under trade name TWEENS®), POLYOXYL® 40 hydrogenated castor oil, fatty alcohol ethoxylates, polyethylene oxide condensates of alkyl phenols, products derived from the condensation of ethylene oxide with the reaction product of propylene oxide and ethylene diamine, ethylene oxide condensates of aliphatic alcohols, long chain tertiary amine oxides, long chain tertiary phosphine oxides, long chain dialkyl sulfoxides, and mixtures of such materials. The nonionic surfactant Poloxamer® 407 is one of the most preferred surfactants because use of Poloxamer helps reduce astringency.

Useful amphoteric surfactants can be broadly described as derivatives of aliphatic secondary and tertiary amines in which the aliphatic radical is a straight chain or branched and wherein one of the aliphatic substituents contains from about 8 to about 18 carbon atoms and one contains an anionic water-solubilizing group, e.g., carboxylate, sulfonate, sulfate, phosphate, or phosphonate. Other suitable amphoteric surfactants are betaines, specifically cocamidopropyl betaine. Mixtures of amphoteric surfactants can also be employed. Preferred levels are from 0.25 to 12 wt%, preferably from 0.5 to 8 wt%, and most preferably from 1 to about 6 wt%.

Sweetening agents

Toothpastes may also contain a sweetening agent. Preferred sweetening agents include sodium saccharin, aspartame, sucralose, thaumatin, acesulfame potassium, stevioside, stevia extract, paramethoxy cinnamic aldehyde, neohesperidyl dihydrochalcone and perillartine. Typical levels are from 0.005 to 5 wt%, more preferably from 0.01 to 1 wt%.

Preferred toothpastes can include one or more bleaching agents such as peroxy compounds e.g. potassium peroxydiphosphate, effervescent systems such as sodium bicarbonate/citric acid systems, colour change systems, and chelating agents from 0.001 to 6 wt%, preferably at from 0.5 to 4 wt%, which include alkali metal salts of citric acid, alanine, glycine and serine.

The most preferred are the alkali metal salts of citric acid, especially potassium citrate and most preferably tri-potassium citrate.

Liposomes can be used to improve delivery or stability of one or more active ingredients. Preferred compositions may also include one or more of breath strips, sparkles, large silica particles, granules, beads, and flavour encapsulates for enhanced sensory benefits or for visual appeal.

Titanium dioxide can also be added to the composition for opacity. Titanium dioxide is generally included from 0.25 to 5 wt%.

It is preferred that the toothpaste compositions contain a flavour. Suitable flavoring components include oil of wintergreen, oil of peppermint, oil of spearmint, clove bud oil, menthol, anethole, methyl salicylate, eucalyptol, cassia, 1-menthyl acetate, sage, eugenol, parsley oil, oxanone, alpha-irisone, marjoram, lemon, orange, propenyl guaethol, cinnamon, vanillin, ethyl vanillin, heliotropine, 4-cis-heptenal, diacetyl, methyl-para-tert-butyl phenyl acetate, and mixtures thereof. Coolants may also be part of the flavor system. Preferred coolants are the paramenthane carboxamide agents such as N-ethyl-p-menthan-3-carboxamide (known commercially as "WS-3[®]") and mixtures thereof. The flavour is generally from 0.001 to 5 wt%.

Physical properties

The viscosity of preferred toothpaste compositions is 80,000 to 500,000 cps, when measured at 1/s and 25 °C with a BROOKFIELD[®] Viscometer using T-bar D-spindle at 5 rpm (revolutions per minute). The viscosity of further preferred toothpaste compositions is 100,000 to 200,000 cps.

Method

In accordance with a second aspect is disclosed a method of promoting oral hygiene comprising a step of applying to the teeth an effective amount of a toothpaste composition of the first aspect.

Use

In accordance with a third aspect is disclosed a composition of the first aspect for use for promoting oral hygiene.

The invention will now be explained with the help of non-limiting exemplary embodiments.

Examples**Example 1: Qualitative descriptive analysis (QDA) of a preferred composition and comparative toothpaste compositions**

A preferred toothpaste composition was made for QDA study. Some comparative compositions were also prepared. The composition of each toothpaste is described in table 1.

Table 1

Ingredient	C-1	C-2	C-3	C-4	X	Y
Sorbitol*	10.0	10.0	15.0	10.0	10.0	10.0
Calcium carbonate	40.0	40.0	40.0	40.0	45.0	50.0
Thickening silica	3.7	3.7	4.5	4.5	2.0	2.0
Veegum® HV	-	-	-	-	0.8	0.8
SCMC	0.7	0.7	0.6	0.6	0.6	0.6
SLS	2.5	2.5	2.5	2.5	2.5	2.5
Water and minors to	100	100	100	100	100	100

Note:

(a) The compositions C1 to C4 are control compositions and X and Y are preferred embodiments of the disclosed invention.

(b) * - this is the actual percentage of sorbitol

A panel of trained expert consumer panelists was convened. Each panel member was asked to use each one of the compositions of table 1. After brushing, each volunteer assigned a score (on a pre-designed scale from 1 to 10) for selected sensory attribute as indicated in table 2. Score of 1 was regarded as the least score and 10 was the maximum score. When all compositions were rated by all panelists, the scores were subjected to statistical analysis. Some consumers were asked to use (and rate) a marketed competitor product having about 20% sorbitol. The statistically analysed data is also shown in tables 2, 3 and 4.

Table 2

Sensory Attribute	C-1	X	Y
Quantity of foam	5.48	5.61	5.57
Creaminess of foam	5.55	5.67	5.62
Flavor intensity while brushing	5.35	5.44	5.48
Cooling during brushing	5.45	5.44	5.46
Mouth freshness after rinsing	5.57	5.64	5.76
Flavour intensity after rinsing	5.12	5.23	5.22

Table 3

Sensory attribute	Marketed competitor product	C-2	C-1	X
Amount of foam	5.58	5.46	5.41	5.54
Creaminess of foam	5.60	5.47	5.44	5.51
Flavour intensity during brushing	5.46	5.41	5.26	5.46
Cooling during brushing	5.54	5.47	5.26	5.59
Cooling after rinsing	5.94	5.58	5.61	5.96
Mouth freshness after rinsing	5.91	5.80	5.60	5.99
Flavour intensity after rinsing	5.21	5.17	4.99	5.23

Table 4

Sensory attribute	C-3	C-4	X
Amount of foam	5.50	5.40	5.51
Creaminess of foam	5.47	5.38	5.48
Flavour intensity during brushing	5.43	5.38	5.43
Cooling after rinsing	5.84	5.80	5.92

Data in table-2 read with table-1 indicates that each preferred composition X and Y was sensorially superior to the control composition C-1 as regards most of the attributes. The material differences in the formulations, as is evident from table-1, are the presence or absence of smectite clay and percentage of thickening silica, both of which were hitherto not known to provide any such benefit, particularly on foam, its creaminess and flavor intensity during and after brushing. Accordingly, the observed technical effects were unexpected considering the known benefits of smectite clay.

Data in table-3 indicates that preferred composition X [notwithstanding its significantly lower humectant content] was sensorially at-par with the marketed competitor product having as high as about 20 wt% sorbitol (on 100% basis). In this case also, the observed technical effects were unexpected having regard to the known benefits of smectite clay and the benefits, if any, brought about by reducing the thickening silica. On the other hand, comparative compositions C-1 and C-2 were again found to be sensorially inferior.

Data in table 4 further confirms the previous findings and reinforces the observations described earlier.

Example 2: Effect of smectite clay on the uptake of antibacterial agent by dental tissue

A preferred toothpaste composition having the formulation as in table 5 was prepared.

Table 5

Ingredient	Wt%
Sorbitol (actual)	10.5
Calcium carbonate	45.0
Thickening silica	2.0
Triclosan	0.3
Veegum	0.8
SCMC	0.4
SLS	2.5
Water and minors	To 100

A panel of forty trained human volunteers was formed for a double-blind, randomized, complete cross-over study. Each panelist was asked to brush the teeth with the composition of table-5. For comparative analysis, each panelist was also asked to brush the teeth with a marketed triclosan containing chalk-based toothpaste which was devoid of smectite clay. Samples of plaque were collected at varying but fixed intervals of time after brushing. Concentration of triclosan in the samples of the plaque was measured by HPLC and the results are shown in table 6.

Table 6

Composition	Concentration of triclosan/ppm					
	After 10 minutes	p value at 95%	After 2 hours	p value at 95%	After 4 hours	p value at 95%
Of table-5	159	0.0063	110	0.0009	48	0.0115
Comparative marketed toothpaste	139		81		37	

Data in table 6 indicates a statistically significant difference (at 95% confidence level) in favour of the preferred composition. The concentration of triclosan at each time interval was significantly higher in the case of the preferred composition. While the actual level of triclosan shows a gradual decline over time which is as expected, what is more important is the observation that the preferred composition of table-5 maintains its superiority as against the comparative marketed toothpaste even after passage of time.

Example 3: Comparative study of antibacterial effect by *in-vitro* method

In this experiment is described a study which compares antibacterial activity of a preferred toothpaste composition. It is measured in terms of availability and retention of triclosan and is compared to a marketed calcium based toothpaste containing 0.3% triclosan.

This effect was studied over a period of four hours using *mutans Streptococci* ATCC 25175 on a HAP (Hydroxyapatite) disc biofilm production model.

A time kill bactericidal approach was adopted. After four hours of incubation of *mutans Streptococci* with saliva-coated and toothpaste-treated HAP discs, the total viable count of organisms which remain attached to the HAP disc were enumerated. The toothpaste which gives the lowest total viable count was regarded as the best.

Table 7 shows the total viable count of *mutans Streptococcus* which are attached to the HAP discs after four hours of incubation and the dentifrice treatment.

Table 7

Product	Exp-1(log₁₀)	Exp-2(log₁₀)	Exp-3(log₁₀)
Control [water]	4.88	4.35	4.37
Marketed competitor toothpaste having 0.3% triclosan	3.73	3.76	3.10
Composition of table 5	2.74	2.48	2.54
Standard fluoridated toothpaste	4.36	3.86	3.75

Data in table 7 indicates that in each of the three experiments, the preferred composition provides lowest total viable count [log₁₀] value thereby indicating that the antibacterial effect of the preferred composition is better than comparative compositions.

Thus, the preferred composition reduces growth of mutants *Streptococci* by a significantly better margin as compared to other toothpastes.

The illustrated examples indicate that a toothpaste composition containing smectite clay and lower amount of thickening silica provide superior sensory profile as well as an increased uptake of antibacterial agent by the oral tissue.

It should be understood that the specific forms of the invention herein illustrated and described are intended to be representative only as certain changes may be made therein without departing from the clear teachings of the disclosure.

Although the invention has been described with reference to specific embodiments, it will be appreciated by those skilled in the art that the invention may be embodied in many other forms

CLAIMS

1. A toothpaste composition comprising:
 - (i) 20 to 60 wt% of a calcium based abrasive;
 - (ii) 3 to 15 wt% humectant; and,
 - (iii) upto 2 wt% thickening silica;wherein said composition comprises 0.2 to 3 wt% smectite clay.
2. A toothpaste composition as claimed in claim 1 comprising 0.05 to 5 wt% antibacterial agent.
3. A toothpaste composition as claimed in claim 2 wherein said antibacterial agent is triclosan.
4. A toothpaste composition as claimed in any one of the preceding claims wherein ratio of said calcium containing abrasive to said smectite clay is in the range of from 1:0.1 to 1: 0.003.
5. A toothpaste composition as claimed in any one of the preceding claims wherein said smectite clay is selected from phyllosilicates, montmorillonites, bentonites, hectorites and derivatives thereof, purified magnesium aluminium silicates, purified sodium magnesium silicates organically modified smectite clay, organically modified montmorillonite clay and mixtures thereof.
6. A toothpaste composition as claimed in claim 5 wherein said smectite clay is magnesium aluminium silicate clay.
7. A toothpaste composition as claimed in any one of the preceding claims comprising 0.1 to 5 wt% cellulosic thickener.
8. A toothpaste composition as claimed in any one of the preceding claims wherein said abrasive is calcium carbonate.

9. A toothpaste composition as claimed in any one of the preceding claims wherein viscosity of said composition is 80,000 to 500,000 cps.
10. A method of promoting oral hygiene comprising a step of applying to the teeth an effective amount of a toothpaste composition as claimed in claim 1.
11. A toothpaste composition as claimed in claim 1 for use for promoting oral hygiene.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2014/055891

A. CLASSIFICATION OF SUBJECT MATTER INV. A61K8/19 A61K8/26 A61K8/34 A61Q11/00 ADD.								
According to International Patent Classification (IPC) or to both national classification and IPC								
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) A61K A61Q Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPO-Internal, WPI Data								
C. DOCUMENTS CONSIDERED TO BE RELEVANT <table border="1"> <thead> <tr> <th>Category*</th> <th>Citation of document, with indication, where appropriate, of the relevant passages</th> <th>Relevant to claim No.</th> </tr> </thead> <tbody> <tr> <td>X</td> <td> WO 2013/007571 A2 (UNILEVER NV [NL]; UNILEVER PLC [GB]; UNILEVER HINDUSTAN [IN]; CHANDRAS) 17 January 2013 (2013-01-17) compositions 3-8 page 5, line 19 - line 27 page 8, line 5 - line 8 page 11, line 9 - line 12 page 14, line 8 - line 12 composition 8; page 14, line 22 page 18, line 26 ----- -/-- </td> <td>1-11</td> </tr> </tbody> </table>			Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.	X	WO 2013/007571 A2 (UNILEVER NV [NL]; UNILEVER PLC [GB]; UNILEVER HINDUSTAN [IN]; CHANDRAS) 17 January 2013 (2013-01-17) compositions 3-8 page 5, line 19 - line 27 page 8, line 5 - line 8 page 11, line 9 - line 12 page 14, line 8 - line 12 composition 8; page 14, line 22 page 18, line 26 ----- -/--	1-11
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.						
X	WO 2013/007571 A2 (UNILEVER NV [NL]; UNILEVER PLC [GB]; UNILEVER HINDUSTAN [IN]; CHANDRAS) 17 January 2013 (2013-01-17) compositions 3-8 page 5, line 19 - line 27 page 8, line 5 - line 8 page 11, line 9 - line 12 page 14, line 8 - line 12 composition 8; page 14, line 22 page 18, line 26 ----- -/--	1-11						
☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.								
* Special categories of cited documents : "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family								
Date of the actual completion of the international search		Date of mailing of the international search report						
21 August 2014		23/09/2014						
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016		Authorized officer Werner, Stefan						

INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2014/055891

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DATABASE WPI Week 201301 Thomson Scientific, London, GB; AN 2012-M28475 XP002721627, & CN 102 579 275 A (HEFEI COLLEGE) 18 July 2012 (2012-07-18) abstract	1-11
A	----- WO 03/030850 A1 (UNILEVER NV [NL]; UNILEVER PLC [GB]; LEVER HINDUSTAN LTD [IN]) 17 April 2003 (2003-04-17) the whole document	1-11
A	----- US 4 081 526 A (ASAKAWA TOSHIRO ET AL) 28 March 1978 (1978-03-28) the whole document -----	1-11

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2014/055891

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 2013007571 A2	17-01-2013	CN 103702652 A EP 2758025 A2 US 2014127143 A1 WO 2013007571 A2	02-04-2014 30-07-2014 08-05-2014 17-01-2013
CN 102579275 A	18-07-2012	NONE	
WO 03030850 A1	17-04-2003	BR 0213608 A CN 1599592 A EP 1432396 A1 HK 1075200 A1 HU 0401682 A2 US 2003072721 A1 WO 03030850 A1 ZA 200401443 A	14-09-2004 23-03-2005 30-06-2004 28-09-2007 28-09-2005 17-04-2003 17-04-2003 10-03-2005
US 4081526 A	28-03-1978	DE 2649449 A1 GB 1502078 A JP S5257339 A JP S5444727 B2 US 4081526 A	18-05-1977 22-02-1978 11-05-1977 27-12-1979 28-03-1978