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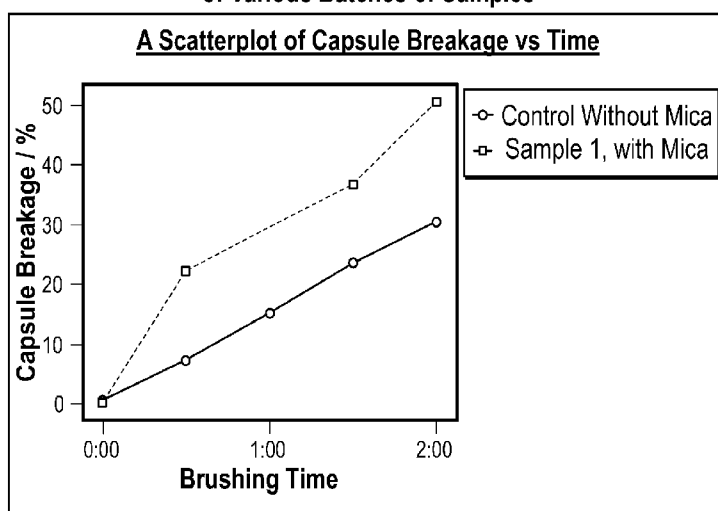
(54) Title: ORAL CARE COMPOSITIONS COMPRISING MICROCAPSULES WITH EFFECTIVE BREAKAGE CHARACTER-
ISTICSA Graph of Brushing Time vs. Capsule Breakage
of Various Batches of Samples

FIG. 2

(57) Abstract: The present invention provides microcapsules with better capsule breakage characteristics, the microcapsules comprising internal phase encapsulating capsule breakage enhancing substances, applications of these microcapsules in oral care, particularly in microencapsulated flavours released during tooth brushing. Accordingly, the capsule breakage enhancing substance is in the size range of 1% to 99% of average microcapsule size most preferably 10% to 30% of the microcapsule size.

ORAL CARE COMPOSITIONS COMPRISING MICROCAPSULES WITH EFFECTIVE BREAKAGE CHARACTERISTICS

FIELD OF THE INVENTION

[0001] The invention relates to microcapsules with enhanced capsule breakage characteristics, the microcapsules encapsulating a capsule breakage enhancing substance material forming an internal phase, wherein the microcapsule are in an average size range from about 5 μm to about 500 μm and the capsule breakage enhancing substance is added in a range from 0.01 to 10% weight of the microcapsule. The invention further discloses applications of these microcapsules in oral care, for example in microencapsulated flavours release during tooth brushing.

BACKGROUND OF THE INVENTION

[0002] US3957964 [assignee: Colgate-Palmolive Company] discloses a dentrifice composition comprising a flavor released from microcapsules.

[0003] US2012/0121669 [assignee: Colgate-Palmolive Company] discloses flavour release from capsules comprising matrix material and plasticizer. Matrix material is selected from chitosan, alginate or agar. Plasticizer is oil, a polymer, or a humectant. However, this patent application does not disclose any material which aids in capsule breakage.

[0004] EP1123695 [assignee: Unilever N.V.] discloses an oral composition comprising capsules containing substantially water insoluble keratin, wherein the keratin is in the diameter no greater than 75 μm .

[0005] While different microcapsules made with plurality of materials forming core and wall, and their breakage releasing plurality of fragrance or flavours while brushing teeth have been disclosed in various applications, there still is need to find a composition which enhances the ease of capsule breakage during brushing.

[0006] Microcapsules must not only perform the functional aspects of holding the desired product to be released effectively; have a pleasant after-feel but also be able to be flexible in breaking.

[0007] Despite the microcapsules proposed by the aforementioned patents and other approaches described in the published literature, there still exists a need for microcapsules to achieve the desired breakage and effective flavour release within the desired time.

[0008] Thus it is always desired to explore more avenues for better microcapsules which impart better breakage and thereby exhibit effective release of actives, fragrances or flavours.

[0009] The inventive microcapsules and their applications in oral care particularly in flavor release in tooth brushing provide all such desired properties.

[0010] We have surprisingly found that varying the amount and using specific size ranges and compositions of mica (coated or uncoated) with respect to microcapsule if included in the encapsulation process can provide the improvement in breakage characteristics of capsule.

SUMMARY OF THE INVENTION

[0011] The present invention provides new and improved microcapsules with better capsule breakage properties. The invention provides microcapsules encapsulating capsule breakage enhancing substances along with other substances forming an internal phase. The invention further provides use of these inventive microcapsules in oral compositions particularly in flavour release during tooth brushing.

[0012] The present invention provides an oral composition comprising microcapsules encapsulating a capsule breakage enhancing material forming an internal phase, wherein the microcapsule are in an average size range from about 5 μm to about 500 μm and the capsule breakage enhancing substance is added in a range from 0.01 to 10% weight of the microcapsule.

[0013] Accordingly, the capsule breakage enhancing substance is in the size range of 1% to 99% of average microcapsule size most preferably 10% to 30% of the microcapsule size.

[0014] The capsule breakage enhancing substance is selected from natural mica, synthetic mica, sericite mica, titanium coated mica, glitter, synthetic fluorophlogopite, borosilicate, glass flakes, metal flakes, talc, kaolin, aluminium oxide (Al_2O_3) platelets, silicon dioxide (SiO_2) platelets, titanium dioxide (TiO_2) platelets, graphite platelet, bismuth oxychloride (BiOCl), calcium borosilicate, synthetic alumina, and boron nitride.

[0015] Desirably, the capsule breakage enhancing substance is mica which includes uncoated mica or mica coated with titanium dioxide preferably in a size range of 1 μm to 495 μm , most preferably 5 μm to 180 μm .

[0016] The present invention provides capsule breakage enhancing substances dispersed in the internal phase which can further include flavor, oil, fragrance, vitamin, dye, /or pigment.

[0017] The internal phase can further comprise excipients and inert materials, binders, fillers, anti-caking agents, disintegrates, colorants, flavorants, odorants, abrasive, surfactant, enzymes, coatings, chelants, preservatives, humectants, diluents, and combinations thereof.

[0018] The present invention provides microcapsules which further encapsulate an oral care active selected from an antibacterial agent, sensitivity agent, tooth-whitening or tooth-bleaching component, anti-calculus composition, anti-plaque agents, anti-gingivitis agents, anti-inflammatory agents, gum protection agents, flavours, colors, and combinations thereof. It is contemplated that the invention can be practiced in conjunction with other oral care actives.

[0019] The microcapsules are distributed in an orally acceptable vehicle, water or a carrier selected from: polysaccharides including alginate, carrageenan, carboxymethylcellulose, chitosan, hydroxyethylcellulose, cellulose ethers, natural gums including xanthan gum, gellan gum, guar gum, guar derivatives, pectin, synthetic rheology modifiers including polyacrylate, polyvinyl alcohol, polyvinylpyrrolidone

[0020] The inventive microcapsules are made up of materials selected from gum arabic, gelatin, casein, albumin, fibrinogen, xanthan gum, soluble collagen peptides, sodium alginate, carboxy methyl cellulose, carrageenan, polyvinyl pyrrolidone, chitosan, alginate and polyvinyl alcohol preferably gelatin and gum acacia.

[0021] The present invention further provides an oral care composition which can be formulated as toothpaste, toothpowder, gel, mouth wash, mouth spray, mouth rinse, lozenges, beads, paints, confectionary and chewing gum.

[0022] In a preferred embodiment, the present invention provides about 30-100% of the microcapsules breakage in less than 2 minutes of brushing.

[0023] The present invention further provides a process for preparing microcapsules by coacervation.

DESCRIPTION OF THE DRAWINGS

An exemplary embodiment of the present invention is illustrated by way of example in the accompanying drawings in which like reference numbers indicate the same or similar elements and in which:

Figure 1 is a graphical presentation of calibration curve of absorbance vs. concentration of dye.

Figure 2 is a graphical presentation of brushing time vs. capsule breakage of various batches of microcapsules.

DETAILED DESCRIPTION OF THE INVENTION

[0024] The prior art suggests that the industry still requires improvements in the field of developing effective cosmetic and especially in personal care compositions particularly those comprising oral-care actives. One of the biggest challenges for oral care composition is to effectively and completely release flavors, fragrance or actives and at the same time, provide a rapid/quick delivery. Not to be bound by the theories, the present invention provides an effective means of meeting the aforementioned unmet need.

[0025] It is believed that the inventive compositions provide effective microcapsules with enhanced breakage ability than in the existing literature, and in this way provides improved and effective oral care compositions. The selection of the particular capsule breakage enhancing material along with specific size ratio with respect to microencapsulate had resulted in high level of breakage achieving 30 to 100% in less than 2 minutes.

[0026] Today's microcapsules must offer not only the functional aspects of flavour release, pleasant after feel, but must also provide flexibility in breakage. Perceivability of a flavour change depends to a larger extent on sufficient level of capsules breakage achievable during brushing.

[0027] The term 'microcapsule' refers to spherically shaped material with a continuous wall surrounding the core. All three states of matter (solids, liquids and gases) may be encapsulated. As used herein for the present invention the microcapsule are in an average size range from about 5 μm to about 500 μm .

[0028] The term 'microencapsulation' refers to the process of surrounding or enveloping one substance within another substance on a very small scale, yielding capsules ranging from less

than 1 micron to several hundred microns in size. The term 'internal phase' refers to that part which may be solid or liquid in which the microcapsule breakage enhancing substance is dispersed and is encapsulated by the microcapsule shell.

[0029] The term 'capsule breakage enhances substance' refers to that substance or combination of substances which bring about a weakening effect of capsules walls and improve level of capsule breakage, wherein the efficiency of the capsule breakage is relative to capsule breakage percentage intended 30% to 100 % in less than 2 minutes. Microcapsules can be made extremely sensitive to pressure by controlling the relative size of capsule breakage enhancing substance and the amount in which it is to be used.

[0030] Microcapsules typically comprise a core surrounded by an encapsulating wall. The inventive capsules comprise an internal phase with capsule breakage enhancing substance.

[0031] In one embodiment, the present invention provides an oral composition comprising microcapsules encapsulating a capsule breakage enhancing material forming an internal phase, wherein the microcapsule are in an average size range from about 5 μm to about 500 μm , preferably from about 5 μm to 250 μm , and more preferably from about 10 μm to 100 μm .

[0032] Particularly, the capsule breakage enhancing substance is selected from natural mica, synthetic mica, sericite mica, titanium coated mica, glitter, synthetic fluorophlogopite, borosilicate, glass flakes, metal flakes, talc, kaolin, aluminium oxide (Al_2O_3) platelets, silicon dioxide (SiO_2) platelets, titanium dioxide (TiO_2) platelets, graphite platelet, bismuth oxychloride (BiOCl), calcium borosilicate, synthetic alumina, and boron nitride.

[0033] In one preferred embodiment of the described invention, the capsule breakage enhancing substance is mica or mica with a titania coating and ranges between the sizes of 1 μm to 495 μm , most preferably 10 μm to 180 μm , most preferably 15 μm to 100 μm . Such mica is incorporated into the shell and the water immiscible internal phase of the microcapsules during their preparation. It has been observed that the presence of mica within the capsules leads to improvements in their ability to rupture upon applying pressure.

[0034] A particularly useful coated form of mica is available commercially from Merck Performance Materials under the brand name of Timiron[®] and from BASF under the brand name Timica[®].

[0035] Particularly, the capsule breakage enhancing substance is added in a range from 0.01 to 10%, more preferably from 1 to 2% w/w of the microcapsule

[0036] According to this invention, the capsule breakage enhancing substance is in the size range of 1% to 99%, more preferably in the size range of 10% to 30 % of average microcapsule size.

[0037] In one preferred embodiment, addition of mica coated titanium dioxide particles to the internal phase leads to a weakening effect of the capsule walls and improves the level of capsule breakage during tooth brushing.

[0038] The present invention provides capsule breakage enhancing substance dispersed in the internal phase which includes flavor, oil, fragrance, vitamin, dye and pigment and combinations thereof.

[0039] The oil can be selected from oil soluble vitamins such as Vitamin A Palmitate, Vitamin E acetate; oil such as mineral oil, sunflower oil, medium chain triglycerides, silicon oil and dimethicone. The pigment can be selected from titanium dioxide, red 30, iron oxides, pigment blue 15 and chromium oxide green. Suitable oil soluble dyes can be readily selected by those skilled in the art.

[0040] Typical agents which may be encapsulated in addition to the capsule breakage enhancing substance forming the internal phase may further include any oral care agent capable of exhibiting a therapeutic, sensory, protective or cosmetic effect and include antibacterial agents, sensitivity agents, tooth-whitening or tooth-bleaching component, anti-calculus compositions, anti-plaque agents, anti-gingivitis agents, anti-inflammatory agents, gum protection agents, flavours, colors, and combinations thereof.

[0041] Accordingly the oral care agents employed in the present invention are common in the art such as:

antimicrobial agents like Triclosan, chlorhexidine, copper, zinc and stannous salts such as zinc nitrate, zinc sulfate, zinc glycinate, sodium zinc citrate and stannous pyrophosphate, sanguinarine extract, metroindazole, quaternary ammonium compounds such as cetyl pyridinium chloride, bis-guanides such as chlorhexidine digluconate, hexetidine, octenidine, alexidine, and halogenated bisphenolic compounds, such as 2,2' methylenebis-(4-chloro-6-bromophenol);

anti-inflammatory agents such as ibuprofen, flurbiprofen, aspirin, indomethacin etc;

anti-cares agents such as sodium and stannous fluoride, amine fluorides, sodium monofluorophosphate, sodium trimeta phosphate and casein;

plaque buffers such as urea, calcium lactate, calcium glycerophosphate and strontium polyacrylates;

vitamins such as Vitamin A, C and E;

desensitizing agents, e.g. potassium citrate, potassium chloride, potassium bicarbonate, potassium oxalate, potassium nitrate and strontium salts;

anti-calculus agents, e.g. alkali-metal pyrophosphates, hypophosphite-containing polymers, organic phosphonates and phosphocitrates etc.;

biomolecules, e.g. bacteriocins, antibodies, enzymes, etc.;

flavours, e.g. peppermint and spearmint oils;

other proteinaceous materials such as collagen;

preservatives; opacifying agents; coloring agents; pH-adjusting agents; sweetening agents;

pharmaceutically acceptable carriers, e.g. starch, sucrose, water or water/alcohol systems etc.;

surfactants, such as anionic, nonionic, cationic and zwitterionic or amphoteric surfactants;

particulate abrasive materials such as silicas, aluminas, calcium carbonates, dicalciumphosphates, calcium pyrophosphates, hydroxyapatites, trimetaphosphates, insoluble hexametaphosphates and so on,

humectants such as glycerol, sorbitol, propylene glycol, xylitol, lactitol etc;

binders and thickeners such as sodium carboxymethylcellulose, xanthan gum, gum arabic etc. as well as synthetic polymers such as polyacrylates and carboxyvinyl polymers;

polymeric compounds which can enhance the delivery of active ingredients such as antimicrobial agents can also be included.

buffers and salts to buffer the pH and ionic strength of the oral care composition; and

other optional ingredients that may be included are e.g. bleaching agents such as peroxy compounds e.g. potassium peroxydiphosphate, effervescing systems such as sodium bicarbonate/citric acid systems, color change systems, and so on.

[0042] The microcapsules are distributed in an orally acceptable vehicle such as water or a carrier selected from: polysaccharides including alginate, carrageenan, carboxymethylcellulose, chitosan, hydroxyethylcellulose, cellulose ethers, natural gums including xanthan gum, gellan gum, guar gum, guar derivatives, pectin, synthetic rheology modifiers including polyacrylate, polyvinyl alcohol, polyvinylpyrrolidone.

[0043] In another embodiment, the present invention provides an oral composition which is formulated as toothpaste, toothpowder, gel, mouth wash, mouth spray, mouth rinse, lozenges, beads, paints, confectionary and chewing gum.

[0044] The present inventive composition provides about 30-100% of the microcapsules breakage in less than 2 minutes of brushing.

[0045] The inventive microcapsules are made up of materials selected from gum arabic, gelatin, casein, albumin, fibrinogen, xanthan gum, soluble collagen peptides, sodium alginate, carboxy methyl cellulose, carrageenan, polyvinyl pyrrolidone, chitosan, alginate and polyvinyl alcohol preferably gelatin and gum acacia. Many suitable encapsulating materials are cross-linked. Cross-linking may be inherent in the encapsulating material or it may be achieved by a cross-linking agent, e.g. glutaraldehyde. In one of the preferred embodiment such microcapsules are available by Ashland under the brand name Captivates[®] encapsulates.

[0046] In another embodiment, the present invention provides a process for preparing microcapsules, wherein the process is called complex coacervation. The cationic and anionic polymers such as gelatin and gum Arabic form a concentrated phase call the complex coacervate. The coacervate exists in equilibrium with a dilute supernatant phase. As water-immiscible core material is introduced into the system, thin films of the polymer coacervate coat the dispersed droplets of core material to form a shell. The microcapsule shells are then solidified to make the capsules harvestable.

[0047] The following non-limiting examples illustrate specific embodiments of the present invention. They are not intended to be limiting the scope of the present invention in any way.

[0048] **Example 1:** Microcapsules with an average size of 50 μm manufactured by the complex coacervation of gelatin and gum acacia followed by crosslinking. Said microcapsules will

possess an internal phase composition of 99% oil-based, water immiscible flavour and 1% titania-coated mica (with a size range of 5 μm to 40 μm). Said microcapsules will be contained at 2% within a toothpaste composition for the purpose of flavour release during tooth brushing.

Example 2: Microcapsules with an average size of 50 μm manufactured by the complex coacervation of gelatin and sodium carboxymethylcellulose followed by crosslinking. Said microcapsules will possess an internal phase of 96% Mineral Oil, 3% D&C Red 30 and 1% titania-coated mica (with a size range of 5 – 40 μm). Said microcapsules will be contained at 2% within a toothpaste composition for the purpose of colour change during toothbrushing.

[0049] Example 3: Method of determining the capsule breakage during tooth brushing

A plastic, grated substrate, 1 cm in width and 10 cm in length was affixed to inside of a centrifuge tube, with the grating facing upwards to act as the substrate for brushing against during the run. Toothpaste samples were prepared using 3% of capsules containing a known concentration of red dye. Toothpaste (1 gm) so obtained was placed onto a toothbrush. Demineralised water [DM] was added to the centrifuge tube and brushing against the plastic grating within the tube was manually carried out for a fixed time period of 30 seconds, 1 minute, 1 minute 30 seconds or 2 minutes. After brushing, the toothbrush was washed into the tube with a small amount of DM water. To extract any free oil from broken capsules, cyclohexane (10 gm) was added and the mixture was manually stirred until an emulsion formed. Following this, potassium chloride (0.1 gm) was added leading to the breakdown of the emulsion and the formation of two immiscible layers. To ensure the removal of impurities from the organic phase, the tube was centrifuged at 3000rpm for 10 minutes. Using UV/Vis spectrophotometer, the absorbance of the organic phase was measured at 499.5nm and compared against a calibration curve (Figure 1) in order to estimate the overall capsule breakage in percent. The calibration curve was prepared using dilutions of dye that were theoretically calculated to reflect various levels of capsule breakage.

[0050] It was acknowledged that a plastic grated surface within a centrifuge tube was not necessarily the best model for a mouth but was sufficient for the aims of the investigation. Ideally, in a full scale study, grated hydroxylapatite disks would be developed to provide a level of action that closer resembles that of *in vivo* tooth brushing.

[0051] Examples of the prepared microcapsule samples used for the investigation were tabulated (Table 1) and are graphically presented in Figure 2. Dye concentrations were converted to theoretical capsule breakage percentages under the approximation that one third of the mass of capsule slurry added to any given toothpaste sample was the oil phase. This approximation holds true assuming that during the preparation of the capsules, 100% of the internal phase added was successfully encapsulated and that no finished product was lost.

[0052] A scatterplot of capsule breakage against time is presented in Figure 2. For the control batch of capsules, that contain no capsule breakage enhancing substance, breakage increases almost completely linearly with brushing time, peaking at 30% breakage after 2 minutes of brushing. The batch of capsules, labelled Sample 1, were very similar to the control batch except that they also contained 1% titania coated mica. The breakage characteristics is significantly improved in comparison to the control batch, peaking at about 50% after 2 minutes.

Table 1: Comparative examples

Batch	Average Diameter	Internal Phase Composition		
		A	B	C
Control (without mica)	40 μ m	99.8 % Mineral oil	0.2% D&C Red No. 17 Dye	-
Sample 1 (with TiO ₂ coated mica)	40 μ m	98.8 % Mineral oil	0.2% D&C Red No. 17 Dye	1.0 % Coated Mica (5-40 μ m)

[0053] The invention has been described in detail with particular reference to preferred embodiments thereof, but it will be understood that variations and modifications can be effected within the spirit and scope of the invention.

We Claim:

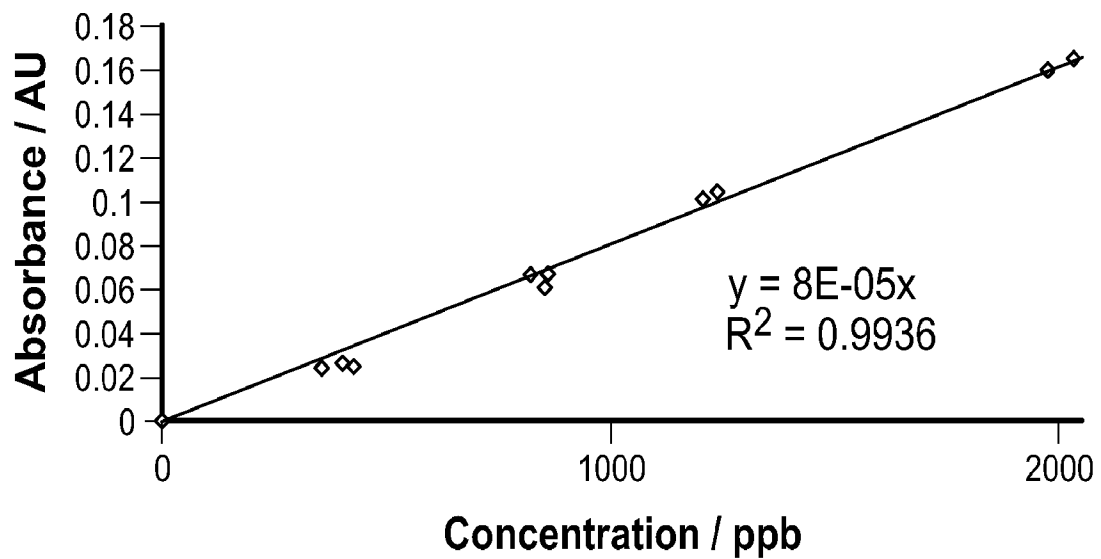
1. An oral care composition comprising microcapsules encapsulating a capsule breakage enhancing substance forming an internal phase, wherein the microcapsule are in an average size range from about 5 μm to about 500 μm and the capsule breakage enhancing substance is added in a range from 0.01 to 10% by weight of the microcapsule.
2. The composition of claim 1, wherein the capsule breakage enhancing substance is in the size range of 1% to 99% of average microcapsule size.
3. The composition of claim 2, wherein the capsule breakage enhancing substance is in the size range of 10% to 30 % of average microcapsule size.
4. The composition of claim 1, wherein the capsule breakage enhancing substance is selected from natural mica, titanium coated mica, synthetic mica, sericite mica, glitter, synthetic fluorophlogopite, borosilicate, glass flakes, metal flakes, talc, kaolin, Alumina (Al_2O_3) platelets, Silicon dioxide (SiO_2) platelets, titanium dioxide (TiO_2) platelets, graphite platelet, (bismuth oxo chloride (BiOCl), calcium borosilicate, synthetic alumina, and boron nitride.
5. The composition of claim 1, wherein the capsule breakage enhancing substance is mica selected from uncoated mica and mica coated with titanium dioxide.
6. The composition of claim 4, wherein the coated or uncoated mica is in the size range of 5 μm to 180 μm .
7. The composition of claim 1, wherein the capsule breakage enhancing substance is dispersed prior to the encapsulation process in the internal phase further including flavor, oil, fragrance, vitamin, dye and pigment .
8. The composition of claim 6, wherein the oil is selected from oil soluble vitamins, Vitamin A Palmitate, Vitamin E acetate, mineral oil, sunflower oil, medium chain triglycerides, silicon oil and dimethicone; the pigment is selected from titanium dioxide, red 30, iron oxides, pigment blue 15, chromium oxide green; and the dye is oil soluble.

9. The composition of claim 6, wherein the internal phase further comprises excipients and inert materials, binders, fillers, anti-caking agents, disintegrates, colorants, flavorants, odorants, abrasive, surfactant, enzymes, coatings, chelants, preservatives, humectants, diluents, and combinations thereof.
10. The composition of claim 1, wherein the microcapsule further encapsulates an oral care active selected from an antibacterial agent, sensitivity agent, tooth-whitening or tooth-bleaching component, anti-calculus composition, anti-plaque agents, anti-gingivitis agents, anti-inflammatory agents, and combinations thereof.
11. The composition of claim 1, wherein the microcapsules are distributed in an orally acceptable vehicle or carrier selected from water or solutions of sodium carboxymethyl cellulose, xanthan gum, hydroxyethyl cellulose.
12. The composition of claim 1, wherein the microcapsules are made up of materials selected from gum arabic, gelatin, casein, albumin, fibrinogen, xanthan gum, soluble collagen peptides, sodium alginate, carboxy methyl cellulose, carrageenan, polyvinyl pyrrolidone, chitosan, alginate and polyvinyl alcohol.
13. The composition of claim 1, wherein the microcapsule shells are made from gelatin and gum acacia or gelatin and carboxymethyl cellulose coacervate complexes.
14. The composition of claim 9, wherein said composition is formulated as toothpaste, toothpowder, gel, mouth wash, mouth spray, mouth rinse, lozenges, beads, paints, confectionary and chewing gum.
15. The composition of claim 10, wherein around 30-100% of the microcapsules break in less than 2 minutes of brushing.
16. The composition of claim 1, wherein the microcapsules are manufactured by coacervation.

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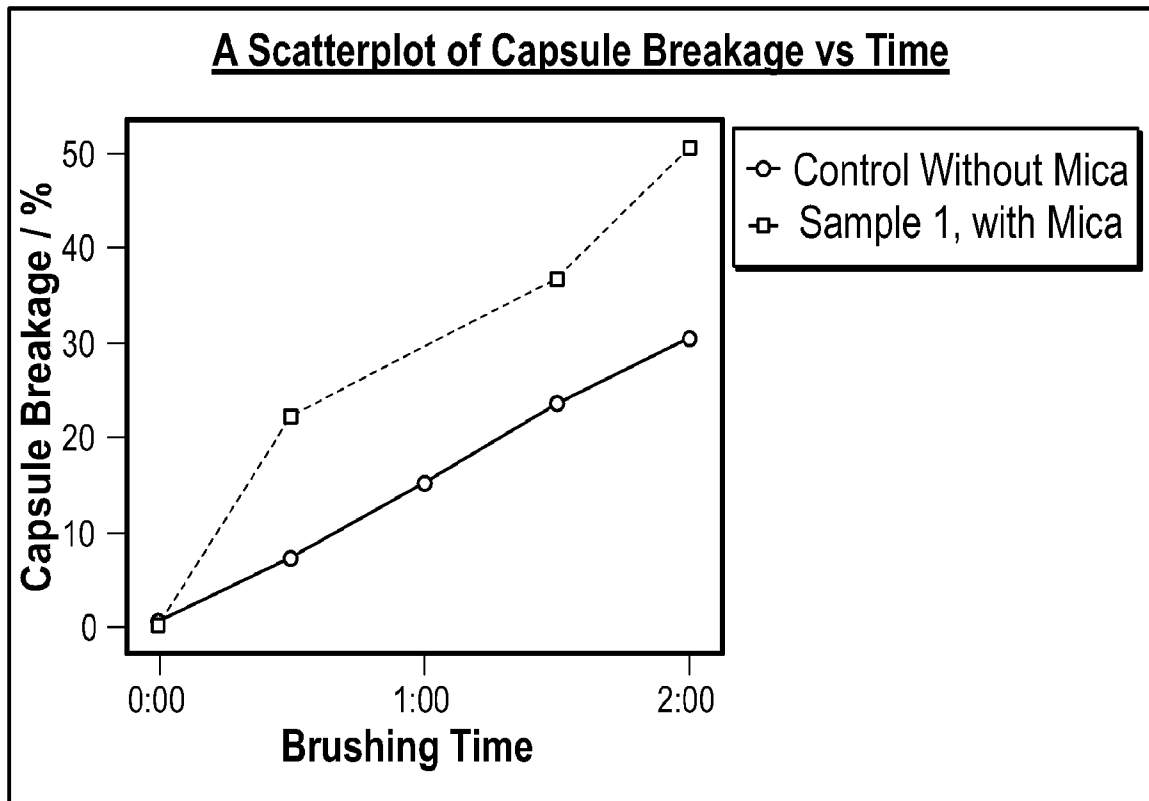
The Calibration Curve of Absorbance vs Concentration of Dye

Absorbance vs. Concentration of D&C Red No. 17 Dye at a Wavelength of 499.5nm.

**FIG. 1**

2/2

**A Graph of Brushing Time vs. Capsule Breakage
of Various Batches of Samples**

**FIG. 2**

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US2015/019439

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - A61K 8/11 (2015.01)

CPC - A61K 8/11 (2015.04)

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)

IPC(8) - A61K 8/00, 8/11, 8/19; A61Q 11/00 (2015.01)

CPC - A61K 8/11, 2800/412; A61Q 11/00 (2015.04) (keyword delimited)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

USPC - 424/49, 451, 452 (keyword delimited)

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

PatBase, Google Patents, Google Scholar.

Search terms used: dentifrice encapsulated oral care mica

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5,700,449 A (KATAYAMA et al) 23 December 1997 (23.12.1997) entire document	1-3, 5, 10-12, 15, 16
Y		4, 6-9, 13, 14
Y	US 4,849,212 A (GLANDORF et al) 18 July 1989 (18.07.1989) entire document	4, 6
Y	US 2002/0159957 A1 (LAGES et al) 31 October 2002 (31.10.2002) entire document	7-9, 13, 14

☐

Further documents are listed in the continuation of Box C.

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* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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"&" document member of the same patent family

Date of the actual completion of the international search

01 May 2015

Date of mailing of the international search report

22 MAY 2015

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