

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO	
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TRA: 11 PCT-PATENTE DE INVENCION CAPITULOS I Y II	FOLIOS: 5
ACT: 519 PRESENTACION	

DIRECCIÓN DE NUEVAS CREACIONES

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Bogotá, D.C.,

Abogado(a)/Señor(a)
JUAN PABLO CADENA SARMIENTO

Asunto: Radicación: 11-84233- -6
 Trámite: 11
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 Folios: 5

Patente de Invención	X			Patente de Modelo de Utilidad	
Vía Nacional					
Vía PCT (Fase Nacional)	X	Capítulo I	X	Capítulo II	
No. Solicitud Internacional	PCT/US2010/021582				
Fecha de Presentación Internacional	21/01/2010				

1. Documentos estudiados

Descripción:	Radicado N°11-08423-00000-0000	Julio 6 de 2011
Reivindicaciones	Radicado N°11-08423-00000-0000	Julio 6 de 2011
	Radicado N°11-08423-00005-0000	Marzo 20 de 2013
Dibujos	Radicado N°11-08423-00000-0000	Julio 6 de 2011
Respuesta solicitante	Radicado N°11-08423-00005-0000	Marzo 20 de 2013
Prioridad:	US 12/356,837	Enero 21 de 2013

2. Análisis de la Prioridad

Se acepta la reivindicación de Prioridad porque esta solicitud fue presentada dentro del plazo establecido (Art. 9 D 486) y su contenido técnico corresponde con el de la prioridad reivindicada.

3. Objeto de la invención

De acuerdo con lo establecido en el numeral 1.2.2.2 del Capítulo Primero del Título 10 de la Circular Única para efectos del examen de patentabilidad, se tendrá en cuenta la tasa pagada por el solicitante al momento de comenzar el examen para las reivindicaciones adicionales; esto es que cuando la solicitud contenga más de diez (10) reivindicaciones y no se haya pagado la tasa complementaria, sólo se estudiarán las cubiertas por la tasa pagada.

Por lo tanto, las reivindicaciones 11 a 21 visibles en las páginas 13 y 14 del radicado N° 11 – 084233 – 00005 – 0000, son aceptadas para ser estudiadas, porque se registra el pago por dichas reivindicaciones adicionales, de acuerdo con los recibos presentes en las páginas 10 y 11 del radicado N° 11 – 084233 – 00005 – 0000.

Título de la solicitud: “Composición de cuidado oral para reducir o eliminar la sensibilidad dental”

El problema técnico consiste en obtener una composición de cuidado para disminuir o eliminarla sensibilidad dental.

Para resolver este problema técnico se presenta una composición dentrífica que contiene un material adherente, partículas de sílice y sales de potasio para la disminución o eliminación de la sensibilidad dental.

El objeto de la presente Solicitud se relaciona con la Clasificación Internacional de Patentes (CIP): A61Q11/00.

4. De la Solicitud de la Patente

Título

Se solicita modificar el título de la presente solicitud, porque al mencionar una composición de cuidado oral, se incluyen muchas posibles formulaciones dentríficas que no hacen parte de la presente invención y por lo tanto, el presente título se considera muy general. Además, se requiere que el título no contenga la expresión uso o términos relacionados el mismo.

Reivindicaciones (Art 30 D486 Art 22 PCT)

El capítulo reivindicatorio no es conciso, porque posee un amplio número de reivindicaciones. Se solicita reducir el número de reivindicaciones, de tal manera que en estas se describa de forma más detallada la posible invención. Además, si el objeto de la presente invención consiste en formulaciones dentríficas para disminuir la sensibilidad dental, se sugiere que dentro del capítulo reivindicatorio se divulguen composiciones dentríficas donde detalladamente se muestren los compuestos activos y auxiliares de formulación específicos en sus respectivas cantidades y proporciones cuantitativas plenamente definidas.

Se menciona nuevamente que el objeto mencionado en las reivindicaciones 1 a 8, donde se mencionan que las partículas de sílice generan un caudal de flujo del 45% o cuando se menciona los valores de tamaño de partícula requerido, el promedio, la mediana, el valor de AUC de la distribución y todos los parámetros estadísticos de la distribución mencionados en las reivindicaciones citadas, son considerados como resultados a alcanzar, porque dicha distribución de tamaños de partícula son consecuencia del proceso de tamizaje. Se solicita modificar estas reivindicaciones, de tal manera que se mencione el producto a patentar más no los resultados a obtener de los mismos.

La reivindicación 9 no es concisa, porque dentro de esta misma, se incluye una cantidad amplia de polímeros que cumplen con la característica de ser material adherente y poseen un peso molecular entre 100.000 y 2.500.000. Se solicita enunciar de manera específica los polímeros que son de utilidad en la presente invención. No es válido remitirse a lo mencionado en la descripción. Dichos polímeros se deben mencionar en la reivindicación

En la reivindicación 15 se mencionan agentes que contiene arginina que no han sido divulgadas en la reivindicación 1. Se solicita realizar la respectiva corrección.

Las reivindicaciones 16 a 18 no son concisas, porque al mencionar un agente antibacteriano, un agente blanqueador, se incluyen muchas sustancias posibles con esta funcionalidad pero no incluidas dentro del objeto de la presente invención. Se solicita mencionar los agentes antibacterianos y blanqueadores específicos.

Es necesario mencionar que aunque un inventor esta en el legítimo derecho de proteger el objeto de su invención en la extensión que este lo decida, el Artículo 30 de la Decisión 486 también define que las reivindicaciones que protegen dicha invención deben ser claras, concisas y estar plenamente soportadas dentro del capítulo descriptivo. De acuerdo a lo anterior, se solicita respetuosamente al solicitante definir el capítulo reivindicatorio de manera más detallada, acorde con las observaciones realizadas anteriormente.

5. Determinación del Estado de la Técnica

El Estado de la Técnica se determinó con base en la fecha de solicitud de prioridad: El 21 de enero de 2009 y se encontraron los siguientes documentos que anticipan el objeto de esta solicitud:

Item	Documento	Título	Fecha de Publicación
D1	US20080267891	“Oral care compositions to reduce or eliminate dental sensitivity”	Octubre 30 de 2008
D2	<i>Journal of dentistry</i> . 2003: 31; 577 – 584	“Occluding effects of dentifrices on dentins tubules”	Noviembre 30 de 2003

El documento D1¹ divulga composiciones dentríficas que contienen partículas de sílice y un material adherente para disminuir o eliminar la sensibilidad dental.

El documento D2² divulga un estudio comparativo de tres composiciones dentrificas ampliamente conocidas para oclusión de la dentina y la efectividad de las mismas

6. Examen de Patentabilidad (Art 14 D486)

Nivel inventivo (Art 18 D486)

La reivindicación 1 divulga una composición para el cuidado oral que comprende: (a) un material adherente; y (b) partículas de sílice en donde la composición para el cuidado oral proporciona (...) y la composición comprende adicionalmente un agente desensibilizador seleccionado de nitrato de potasio, cloruro de potasio y citrato de potasio” (Ver página 13 del radicado N° 11-084233-00005-0000)

Comparación de las características técnicas **esenciales**, con las del Estado de la Técnica:

Solicitud	D1	D2
Composición para el cuidado oral	Composición dentrífica para el cuidado oral	Composiciones dentríficas para la sensibilidad dental

1 http://worldwide.espacenet.com/publicationDetails/originalDocument?CC=US&NR=2008267891A1&KC=A1&FT=D&ND=3&date=20081030&DB=worldwide.espacenet.com&locale=es_LP

2 http://ac.els-cdn.com/S0300571203001155/1-s2.0-S0300571203001155-main.pdf?_tid=65600d6c-bc9d-11e2-aeef7-00000aab0f27&acdnat=1368539677_a5ba26c40a53a08670dd2b5750c0ef8e

Los Estados de la Técnica D1 y D2 se consideran los más cercanos a la invención definida en la reivindicación 1 porque divulgan composiciones dentríficas para disminuir o eliminar la sensibilidad dental que contienen materiales adherentes, partículas de sílice y sales de potasio (D1, Pág. 3, párrafo [0029], tabla 1, composiciones C y D; Pág. 4, reivindicaciones 1 a 26; D2: página 579, tabla 1).

La diferencia entre la invención, definida en la reivindicación 1 con respecto a las composiciones descritas en D1 y D2 consiste en la composición definida en la presente solicitud contiene como agente desensibilizador alguna sal de potasio, mientras que las composiciones descritas en D1 no contienen ninguna sal de potasio. Las composiciones descritas en la invención describen partículas de sílice cuya distribución de tamaño de partícula oscila entre 2 y 5µm, mientras que en D2 se describe que las partículas de carbonato de magnesio poseen un tamaño de partícula de 4 µm (D2: Pág. 580, descripción figuras 12 y13).

Estas diferencias al parecer no conceden algún efecto técnico ventajoso o inesperado a las composiciones de la presente invención con respecto a lo divulgado en D1 y D2, porque las composiciones descritas en D1 poseen un menor índice o porcentaje de flujo a través de la dentina con respecto a la composición descrita en la presente invención (Composición E). Para D1, el porcentaje de flujo a través de la dentina de las composiciones C y D son del 22% y 5% respectivamente, mientras que la composición E descrita en la solicitud posee un porcentaje de flujo del 31% (ver en la solicitud el folio 36, tabla 5; ver en D1 la página 3, párrafo [0033]). Además, D2 divulga composiciones dentríficas ampliamente conocidas que generan valores de porcentajes de oclusión de los túbulos de la dentina mayores al 80%, que superan al divulgado en la presente solicitud para la composición E (75% de oclusión), particularmente la composición Emoform que contiene sales de potasio, genera un porcentaje de oclusión del 96% y la composición Sorriso que contiene partículas de sílice (silicato de sodio), genera un porcentaje de oclusión del 98%, de acuerdo con la tabla divulgada en D2 (ver en D2 pág. 579, tabla 2).

La sociedad solicitante considera que la presente solicitud es inventiva porque la composición para el cuidado oral que contiene partículas de sílice entre 2 y 5µm y una sal de potasio, reduce la sensibilidad dental mediante dos mecanismos: (i) desensibilización de los nervios mediante la entrega de iones potasio a través de la dentina, y (ii) la oclusión de los túbulos de dentina por las partículas de sílice, dando lugar a que la combinación de sales de potasio con partículas de sílice en

presencia de un material o polímero adherente, logran el sorprendente efecto técnico de producir un 75% de la oclusión de la dentina.

Con respecto al argumento anterior, D2 divulga composiciones dentríficas ampliamente conocidas en el Estado de la Técnica que generan un efecto técnico mucho mejor que el propuesto en la presente invención, ya que en estas composiciones no se requiere la combinación de las sales de potasio con partículas de sílice para generar un óptimo efecto desensibilizador dental. Inclusive, D2 sugiere que la presencia de partículas de sílice en composiciones dentríficas tiene el efecto de oclusión de la dentina (ver en D2 la página 583, columna 1, líneas 4 a 11). Además, el efecto técnico que sugiere D2 para obtener mejores porcentajes de oclusión consiste en la precipitación de diversas sales a través del lumen de los túbulos de dentina (ver en D2 la página 583, columna 2, líneas 2 a 4) .

El problema técnico objetivo que pretende resolver esta invención se puede formular así: ¿Cómo modificar las composiciones divulgadas en D1 para obtener composiciones alternativas para el cuidado oral que además de contener un material adherente y partículas de sílice con tamaños de partícula entre 2 y 5µm, también contengan sales de potasio para disminuir o eliminar la sensibilidad dental?.

D1 divulga en la página 2, párrafo [0021] que las composiciones descritas también pueden contener algún agente desensibilizador, entre los cuales se divulga el nitrato de potasio y menciona que las partículas de sílice poseen un tamaño menor de 8µm (D1: Página 1, ver el abstract). D2 menciona en la página 580, descripción figuras 12 y 13 que las partículas de carbonato de magnesio poseen un tamaño de partícula de 4 µm.

Por lo tanto, una persona medianamente versada en la materia y conocedora de los conceptos divulgados en D1 y D2, lograría adicionar una sal de potasio divulgada en D2 y basarse en los tamaños de partícula de los componentes de D2 a las composiciones C y D anteriormente descritas en D1 y obtener partículas de sílice menores a 8µm e incluso, de 4 µm para optimizar la capacidad desensibilizadora dental, obteniendo de esta forma, las composiciones dentríficas propuestas en la presente invención de manera obvia o evidente.

En consecuencia, la reivindicación 1 de acuerdo a lo divulgado en D1 y D2, carece de Nivel Inventivo.

Las reivindicaciones 2 a 21, dependientes de la reivindicación 1, no divulgan características inventivas adicionales a las composiciones de la reivindicación 1, porque al revisar el capítulo reivindicatorio en la página 4 de D1, se observa que las características adicionales mencionadas en las reivindicaciones de la presente invención ya se reclaman en las reivindicaciones de D1. Por lo tanto, estas reivindicaciones también carecen de Nivel Inventivo.

7. Conclusión

Los requisitos de Patentabilidad de la presente Solicitud están afectados así:

Item	Documento	Reivindicaciones afectadas	Requisito que afecta
D1	US20080267891	1 a 21	Nivel inventivo
D2	<i>Journal of dentistry</i> . 2003: 31; 577 – 584	1 a 21	Nivel inventivo

En cumplimiento del artículo 45 de la Decisión 486 de la Comisión de la Comunidad Andina, se le indica que debe dar respuesta dentro del término de sesenta (60) días contados a partir de la fecha de notificación de la presente comunicación. En caso de no dar respuesta al presente requerimiento, o si a pesar de la respuesta subsistieran los impedimentos para la concesión, se denegará la patente.

Se invita al solicitante se sirva indicar, si así lo considera, su renuncia al término faltante de sesenta (60) días para dar respuesta a este requerimiento y presentarla por escrito.

Finalmente, se le recuerda al solicitante que si como respuesta a este requerimiento llegara a modificar el capítulo reivindicatorio y éste contenga más de diez (10) reivindicaciones, no pagará la tasa de modificación voluntaria pero sí la correspondiente al pago por reivindicación adicional.

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



JOSÉ LUIS SALAZAR LÓPEZ
Director de Nuevas Creaciones (c)

El anterior requerimiento fue notificado por fijación en lista, de fecha 2013-05-28

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ELSEVIER

Occluding effect of dentifrices on dentinal tubules

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KEYWORDS

Hypersensitive dentin;
Dentifrices; Occluding
tubules; Scanning
electron microscopy

Summary Objectives: The objective of this study was to evaluate the tubule occluding ability of three commercial available dentifrices (Sensodyne, Emoform and Sorriso) by Scanning Electron Microscopy.

Methods: Fifty cervical areas from buccal and lingual surfaces of sound third human molars were used. Cervical enamel was wet abraded to expose flat dentin surfaces and further polished with diamond pastes. Specimens were randomly divided into five groups ($n = 10$): G1—no brushing; G2—brushing without dentifrice; G3—brushing with Sensodyne; G4—brushing with Emoform; G5—brushing with Sorriso. Brushed specimens were treated for 4 min per day, for 7 days in a toothbrushing machine. Specimens were prepared and observed under SEM for calculation of the percentage of occluded tubules. In addition, slurries of toothpastes were analyzed by X-ray microanalysis. Data were statistically analyzed by ANOVA and Tukey test ($p < 0.05$).

Results: Means of occluded tubules in descending order were: G5— $98.83 \pm 3.31\%$ (a), G4— $96.02 \pm 5.24\%$ (a), G3— $80.12 \pm 24.65\%$ (a), G1— $37.31 \pm 24.22\%$ (b); G2— $22.92 \pm 15.19\%$ (b). The three tested dentifrices produced increased dentinal occlusion as compared to controls ($p < 0.05$) but equivalent occlusion among each other. Calcium from calcium-carbonate abrasive was identified in all dentifrices.

Conclusions: Results indicated that the use of all dentifrices occluded tubules more than no brushing and brushing without dentifrices groups. Thus, the tested dentifrices seem effective for desensitization by tubule occlusion.

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Introduction

Physiological and morphological studies have demonstrated that dentine hypersensitivity in cervical areas results from open dentinal tubules at the surface.^{1–3} According to the hydrodynamic mechanism, exposed dentin with patent tubules

allows the movement of tubule fluid leading to dentine sensitivity.⁴ A viable treatment modality consists of desensitization by tubule occlusion, which decreases both dentine permeability and fluid movement, thereby reducing dentine sensitivity.¹

Even though the hydrodynamic theory is the currently accepted theory for transmission of stimuli in dentine,^{5–8} other possible mechanisms have been suggested, such as the transducer and the modulation theories. Dentine hypersensitivity

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has been treated by a number of agents, which have been claimed to reduce pain by occluding dentinal tubules. However, other possible modes of action for desensitizing agents have been proposed, such as coagulating or precipitating constituents of tubular fluids, or by stimulating the formation of secondary dentine.^{9,10}

Various chemical agents have been used for dentinal tubules occlusion and for dentine hypersensitivity treatment.^{1,11,12} Desensitizing toothpastes contain some active agents, such as fluorides, strontium salts and potassium. Moreover, toothbrushing is generally performed with dentifrices containing abrasives that can increase tubule occlusion.^{5,13,14} This additional blockage may occur by directly occluding the tubules with abrasive or indirectly by the formation of a smear layer during toothbrushing.^{6,15,16}

Although dentifrice abrasives, such as calcium carbonate, alumina, dicalcium phosphate, silica and diatomaceous earth, have therapeutic potential for partially or completely obturating tubules,^{14,15} other reports investigated the effect of brushing on dentine abrasion with the use of non-abrasive dentifrices.^{17,18} It is unclear whether the active ingredient alone or in combination with abrasive can occlude the dentin tubules. While studies indicate that chemical agents exert a significant clinical effect, other toothpaste ingredients also seem to participate in tubules occlusion.^{13,14,19}

In vivo studies have shown considerable decrease in hypersensitivity when teeth were brushed with desensitizing dentifrices.^{15,16,20} However, the problem arises when attempts are made to determine the mode of action of active agents incorporated into these dentifrices. The aim of this study was to evaluate the effects of desensitizing dentifrices on dentinal tubule occlusion by scanning electron microscopy (SEM). In addition, X-ray microanalysis was used to identify slurry dentifrice components that are related to hypersensitivity management.

Materials and methods

Specimens were obtained from twenty-five extracted sound human third molars stored in 10% formalin (pH 7.0) at room temperature for no longer than two months prior to their use.

Dentin specimen preparation

The teeth were mesio-distally sectioned using a water-cooled diamond saw (KG Sorensen, Barueri,

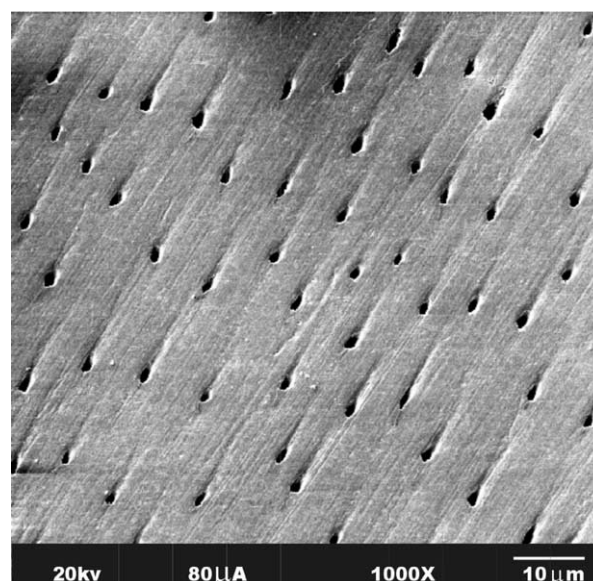


Figure 1 Flat control dentine prepared with diamond pastes that received no treatment. Note absence of smear layer over surface and open dentinal tubules.

SP, Brazil) to obtain fifty buccal and lingual surfaces. Each surface was wet ground (1000- and 1200-grit aluminum oxide abrasive paper) on a polishing machine to remove enamel and to expose a flat underlying dentine surface in the cervical area. One block was obtained from each flat cervical dentine surface, measuring 5 × 5 × 3 mm. The exposed dentine surfaces were polished with 6, 3, 0.5 and 0.25 µm diamond pastes and the specimens were ultrasonicated in distilled water for 12 min to remove residual smear layer and to open dentinal tubules, simulating hypersensitive cervical regions (Fig. 1).

Experimental groups and treatments

Specimens were randomly divided into five groups, each with ten specimens:

G1: Specimens were immersed in artificial saliva²¹ for one week. The artificial saliva contained (mmoles/l): Distilled water (700 ml), CaOH₂ (1.56 mM), KCl (150.00 mM), HCl (36.00 mM), H₃PO₄ (0.88 mM), buffer (99.7 mM), pH 7.2.

G2: Specimens were brushed with distilled water, for 2 min twice per day for 7 days.

G3, G4 and G5: Specimens were brushed with Sensodyne, Emoform and Sorriso, respectively, for 2 min twice per day for 7 days.

Compositions of dentifrices used in this study are summarized in Table 1. Specimens from G3, G4 and G5 were brushed with the dentifrice slurries, which were prepared by diluting 2 g of the dentifrice in 6 ml of distilled water. Nylon multi-tufted

Table 1 Toothpastes used in the study.

Toothpaste	Manufacturer	Active Ingredients
Sensodyne	Stafford-Miller, Ind. Ltda., Rio de Janeiro, RJ, Brasil	Strontium chloride 10%, titanium dioxide, calcium carbonate
Emoform	Byk Química e, Pharmaceuticals Ltda., Diadema, SP, Brazil	Potassium nitrate, calcium carbonate, magnesium carbonate, sodium chloride, sodium sulfate, potassium sulfate, sodium bicarbonate, sodium lauryl sulfate
Sorriso	Kolynos do Brazil Ltd., São Bernardo do Campo, SP, Brazil	Sodium monofluoro-phosphate (1500 ppm Fluoride), calcium carbonate, sodium lauryl sulfate, sodium silicate

toothbrushes (Oral B Soft, Oral B, Manaus, AM, Brazil) were used, and brushing was performed using a toothbrushing machine (Equilabor, Piracicaba, SP, Brazil), at constant loading (200 g) for 250 strokes/minute. After each brushing session, specimens were washed with distilled water, kept in artificial saliva and stored in a shaker machine (TE 420-Tecnal, Piracicaba, SP, Brazil) at $37 \pm 1^\circ\text{C}$.

SEM analysis

After the last brushing session, specimens were stored in a shaker machine for 2 h, washed with distilled water and sputter coated (MED 010-Balzers, Balzers, Leichtenstein) with a thin gold layer. Photomicrographs were taken (DSM 940-A-ZEISS, Oberkochen, Germany) from each dentinal surface examined, approximately 100 μm below the cemento-enamel junction, at $1000\times$ magnification. Percentage of occluded tubules was obtained by dividing the total number of occluded tubules by the total number of tubules in each photomicrography. This result was then multiplied by 100, to obtain the percentage of occluded tubules for each photography. Results were statistically analyzed by one-way Analysis of Variance (ANOVA) and Tukey test ($p < 0.05$).

X-Ray microanalysis

Slurries of all dentifrice were submitted to X-ray microanalysis. A drop of slurry was dispensed on a stub that was kept at 80°C for 40 min, to complete water evaporation. Dried slurries were coating with a thin carbon layer (MED 010-Balzers, Balzers, Leichtenstein) and examined by energy dispersed X-ray detection system (Link ISIS-Oxford) connected with a SEM.

Results

Table 2 displays the mean percentage of occluded tubules in each group. The groups brushed with dentifrices showed a significantly higher percentage

of occluded tubules than control groups (G1 and G2). All tested dentifrices produced almost 100% of occluded tubules, but there was no significant difference among them. Specimens treated with Sensodyne, Emoform and Sorriso presented the following percentage of occluded tubules, respectively: 80.1 ± 24.7 ; 96.0 ± 5.2 and 98.8 ± 3.3 . Saliva immersion (G1) resulted in $37.3 \pm 24.2\%$ of dentinal tubule occlusion while specimens brushed without dentifrice (G2) produced $22.9 \pm 15.2\%$ tubule occlusion. No significant difference was noted between the control group and the group that was brushed without dentifrice.

Figs. 2 and 3 show typical untreated dentin surface. Both treatments resulted in reduction of the tubule lumen diameters (arrow) and complete obliteration of some tubule openings (arrowhead). Specimens brushed without dentifrice produced no alterations on percentage of occluded tubules, when compared to specimens only stored in artificial saliva. A rough dentin surface with toothbrush scratches was noted for dentifrice brushed specimens (Figs. 4-6). Different modes of obliteration and deposits over dentine surface were observed for groups brushed with dentifrices. Sensodyne and Sorriso groups were characterized by deposition of crystal-like and granular structures on the dentin surface and in the lumen of the tubules (Fig. 4 and 5). Dentin surfaces treated with Emoform dentifrice exhibited occluded tubules by deposition of a thin amorphous layer over the dentin (Fig. 6).

Table 2 Average numbers of occluded tubules per 100 dentinal tubules on the dentin surface after brushing with dentifrices and without dentifrice.

	Mean (%) $\pm$ SD
Sorriso	$98.8 \pm 3.3a$
Emoform	$96.0 \pm 5.2a$
Sensodyne	$80.1 \pm 24.7a$
Control	$37.3 \pm 24.2b$
Brushed without dentifrice	$22.9 \pm 15.2b$

Mean $\pm$ SD ($n = 10$). Groups identified by different letters are significantly different ($p < 0.05$).

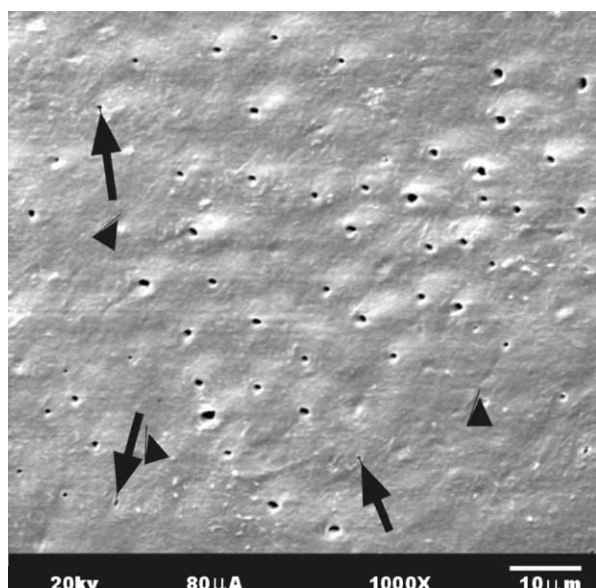


Figure 2 Control dentine kept in artificial saliva without brushing after seven days storage. Arrows indicate tubules with reduction of tubule lumen diameters and arrowheads show occluded tubules.

Some elements, including phosphorus (P), silicon (Si), chlorine (Cl), sodium (Na) and magnesium (Mg), were detected, but the most predominant element was calcium (Ca) (Fig. 7A-C). Fig. 8 shows dentine treated with Sorriso, which presented similar crystalline deposits detected in all dentifrice-dried slurries. Crystalline deposits were investigated (Fig. 9 and 10) and X-ray analysis indicated that crystals

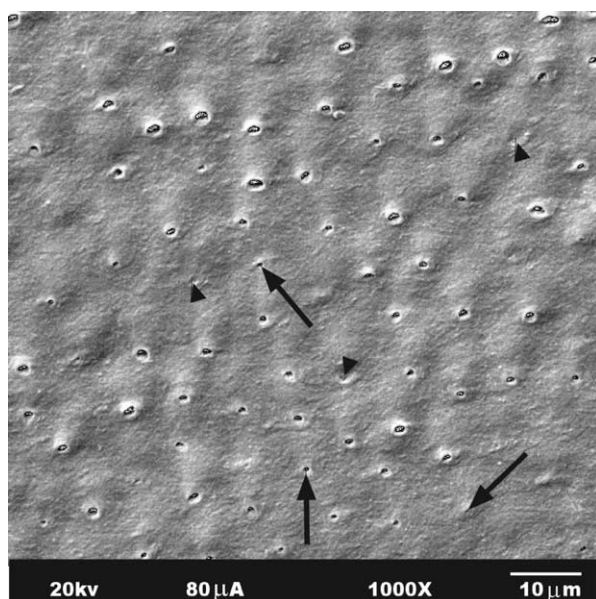


Figure 3 Dentine brushed with artificial saliva but without dentifrice. Arrows indicate tubules with reduction of lumen diameters and arrowheads show occluded tubules.

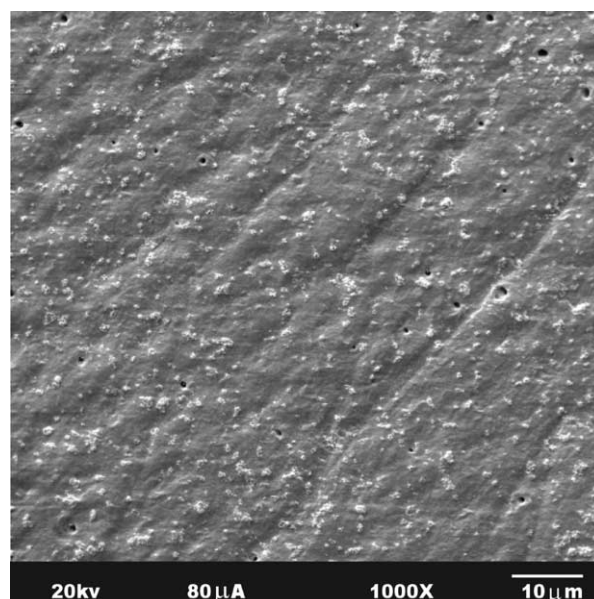


Figure 4 Dentine brushed with Sensodyne. Deposition of crystals-like and granular structures can be noted on treated dentin surface.

contain calcium (Fig. 11). Emoform slurry examination revealed Mg-containing particles approximately 4 μm in diameter (Figs. 12 and 13).

Discussion

In order to confirm that this study was performed in superficial dentin, simulating dentin hypersensitivity (Fig. 1), the tubule density of specimens was

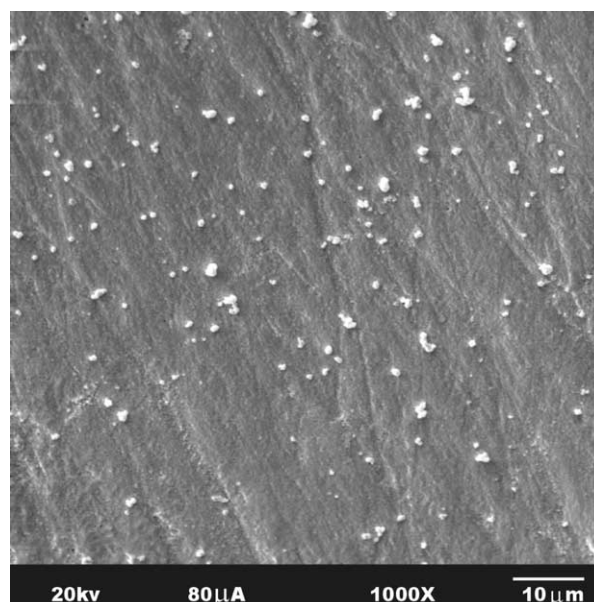


Figure 5 Dentine brushed with Sorriso. Deposition of granular structures can be noted on treated dentin surface.

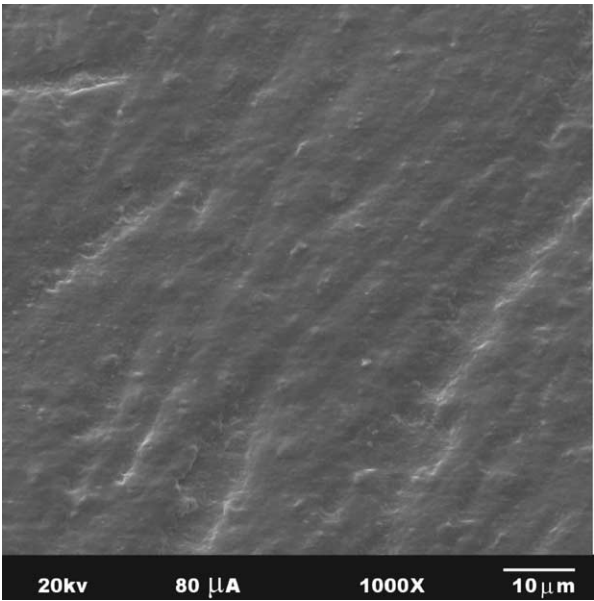


Figure 6 Dentine brushed with Emoform. A precipitation layer of precipitates can be noted on treated dentin surface.

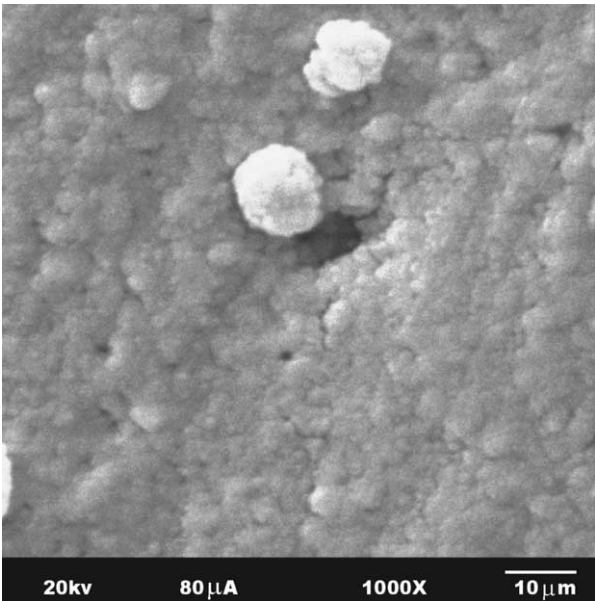


Figure 8 Crystal deposited in the tubule lumen of dentin surface with Sorriso dentifrice.

compared to previously reported values.²²⁻²⁵ Studies showed that superficial dentin has from 17,000 to 20,000 tubules/mm². The dentinal surfaces investigated in the present study showed similar tubule density, confirming that this study was performed in superficial dentin. For hypersensitive areas, approximately 75% of dentin tubules are opened, when inspected under SEM. Non-sensitive cervical dentin show only 24% of opened tubules.³

Specimens brushed with dentifrice exhibited higher percentage of tubule occlusion than G1 and

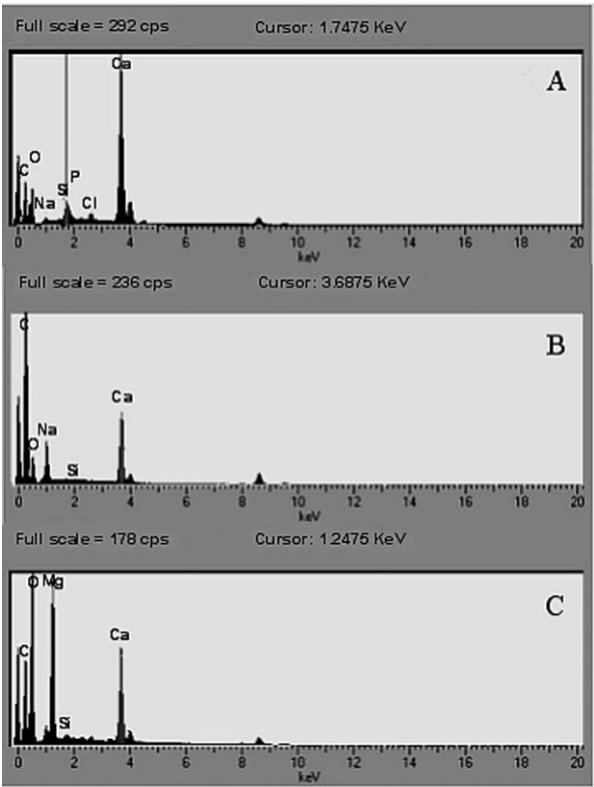


Figure 7 Surface scan of dentifrice slurry. Elements identified by energy dispersive X-ray spectroscopy micro-analysis of Sensodyne (A), Sorriso (B) and Emoform (C) slurries.

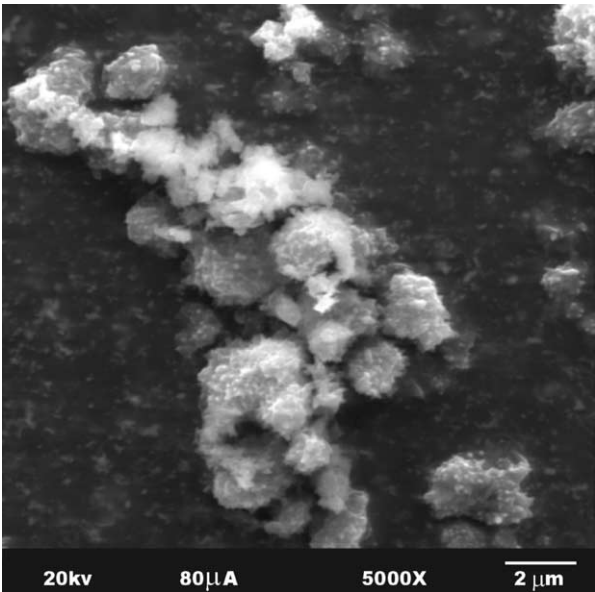


Figure 9 Crystal like-structures commonly observed in slurry dentifrices.

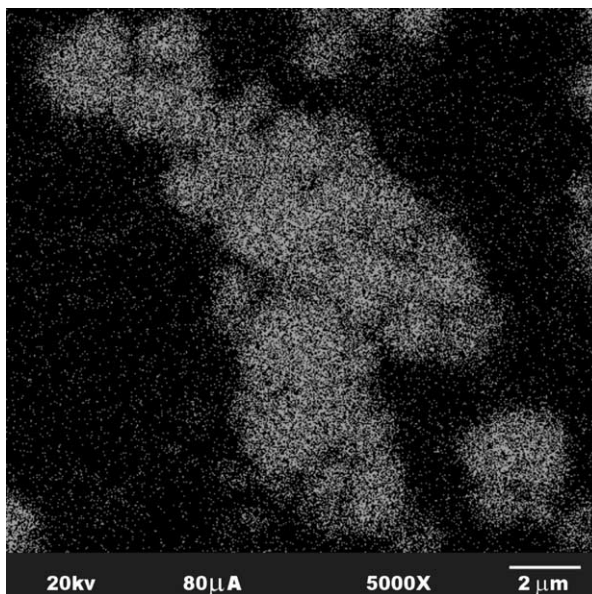


Figure 10 Elemental dot-map showing distribution of calcium in crystal like-structures in Fig. 9.

G2 control groups. Specimens immersed in artificial saliva (G1) or brushed without dentifrice (G2) were not significantly different. For these specimens, dentinal tubule occlusion can only be attributed to calcium phosphate precipitation on the dentin surface, because artificial saliva is supersaturated with respect to hydroxyapatite.²¹ Despite a reduction in the tubule lumen and complete occlusion of some tubules (Figs. 2 and 3), specimens from control groups still had most of the tubular orifices opened.

In this study, three toothpastes with the same abrasive component were selected to analyze

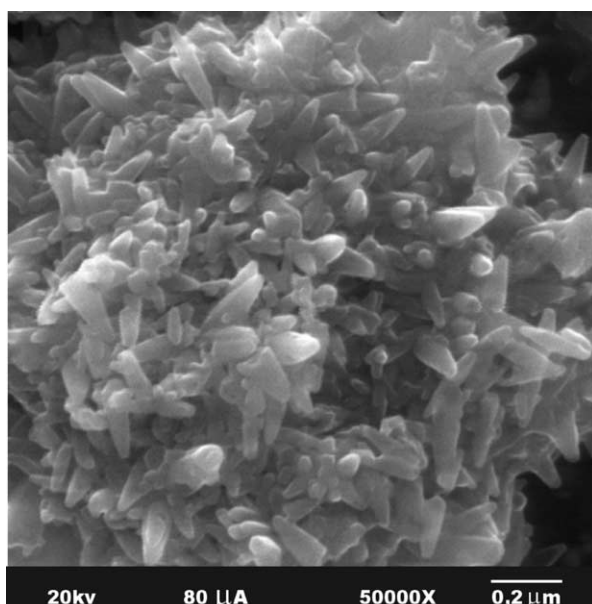


Figure 11 Crystal structures at higher magnification.

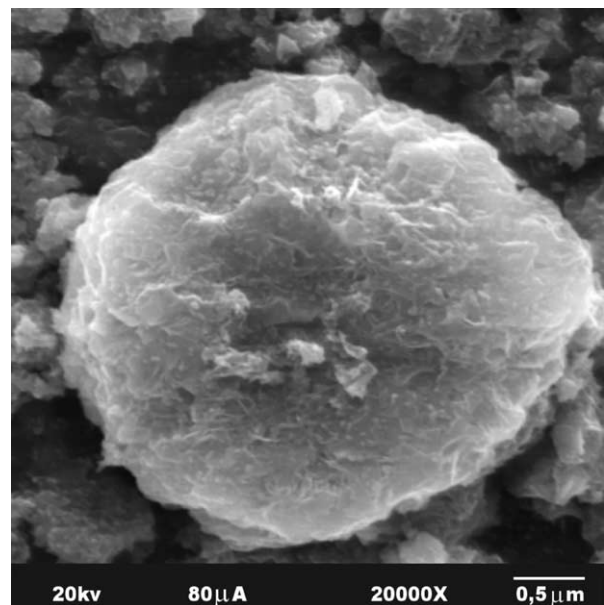


Figure 12 Particle observed in Emoform slurry. Note that it is larger than the surrounding particles.

the occluding effect of desensitizing dentifrices. Studies have shown that abrasives can present different abrasion properties and produce tubular occlusion with varied degrees of success.^{26,27} Thus, three commercial available dentifrices were analyzed: Sensodyne, Emoform and Sorriso, all of which contain calcium carbonate as the abrasive agent and strontium chloride, potassium nitrate and sodium monofluoro-phosphate as active ingredients, respectively.

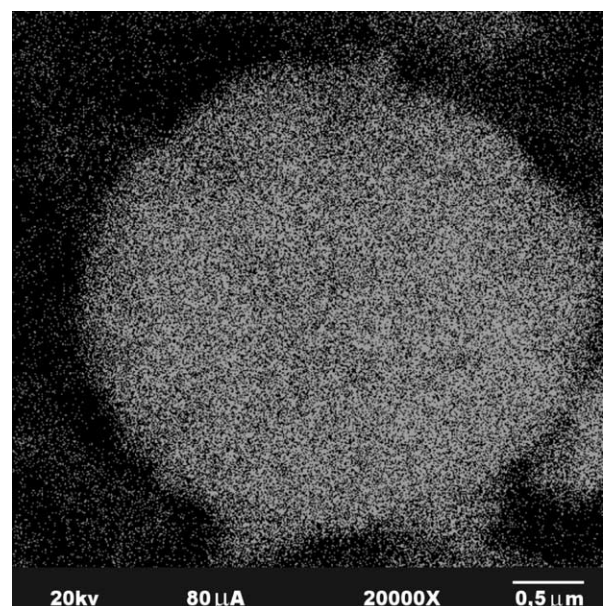


Figure 13 Elemental dot-map showing the presence of magnesium within the particle in Emoform slurry shown in Fig. 12.

Although manufacturers only mentioned the presence of calcium carbonate as abrasive agents, X-ray analyses detected silicon in all dentifrice slurries (Figs. 7). The Sorriso dentifrice label lists the presence of sodium silicate in its composition; however, this component is not an abrasive. Tubule occluding particles have been reported to be silica in specimens brushed with silica-containing dentifrices.¹⁴ Therefore, if dentifrices contain silica, occluding effects can also be attributed to its presence. Calcium was identified in all dentifrice slurries (Fig. 7). Particles containing cluster of crystals were often noted in slurries under SEM examination (Fig. 9 and 11) and X-ray analysis showed that particles contained calcium (Fig. 10). Dentine specimens that were brushed with dentifrices showed crystal particles occluding tubules and deposited on the dentine surface (Figs. 4, 5 and 8), similar to the crystals found in dentifrice slurries (Fig. 9). Thus, calcium carbonate abrasive probably contributed to the superficial occlusion of dentinal tubules. Nevertheless, the presence of calcium on the dentin surface could not be easily confirmed because of the background noise produced by mineral content of the underlying dentine.

Large particles were observed in the Emoform slurry (Fig. 12) and energy dispersed X-ray microanalysis revealed that magnesium was a component of the particle (Fig. 13). Around the particles containing magnesium, smaller particles containing abrasive components were noted. Sodium and phosphorus were detected in Sensodyne and Sorriso slurries; however, X-ray microanalysis was not able to identify important elements from active ingredients like fluoride, strontium and potassium. Sorriso dentifrice presents several sources of sodium, and the presence of phosphorus could be related to sodium monofluoro-phosphate active component containing 1500 ppm of fluoride. In the Sensodyne slurry, the presence of chloride indicates its active ingredient strontium chloride. Although there is no evidence of tubule occlusion by strontium in this study, this active ingredient may act on the interruption of neural transmission.²⁸ Fluoride blocks dentinal tubules through precipitation,²⁹ while potassium is a chemical agent that provides a physiological mechanism for desensitizing action.³⁰ The mechanical desensitization by tubule occlusion with the active ingredients was not confirmed, because active ingredients were not the main components deposited over dentine surface and in the dentin tubules.

Even though no significant difference in the percentage of occluded tubules was detected among the three tested dentifrices, each appeared

to promote different modes of tubule obliteration. Sensodyne and Sorriso promoted the occlusion of tubules by depositing granular and crystal-like structures within lumen of the tubules (Fig. 8). The high level of calcium in these structures, and their morphological characteristics, suggest that they consist of calcium carbonate. Emoform exhibited the same ability to occlude tubules; however, its use resulted in a deposition of a thin amorphous layer. The slurry observed under SEM exhibited a greater number of particles (Fig. 12) than Sensodyne and Sorriso slurries. Therefore, the composition of this layer may comprise the intense precipitation of carbonates particles such as calcium and magnesium carbonate. However, the abrasivity of the dentifrice may create a thin smear layer that also occludes the tubules.

Dentifrice active ingredients seem to result in few surface deposits. In contrast, the abrasive system of dentifrices formed granular and crystal deposits or a thin layer over dentin surface that partially or completely occluded tubules. SEM and X-ray analysis provide evidence that the tested dentifrices may produce their desensitizing responses by tubule occlusion. However, further research is necessary to verify if the abrasive particles alone have the potential of blocking the hydrodynamic mechanism, which produces dentin hypersensitivity. Confirmation of SEM results using quantitative methods such as changes in hydraulic conductance of dentine should be done.^{31,32}

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