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22. SANTAFE DE BOGOTA, D.C.

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☐ Patente de Modelo de Utilidad.

☐ Capítulo I.

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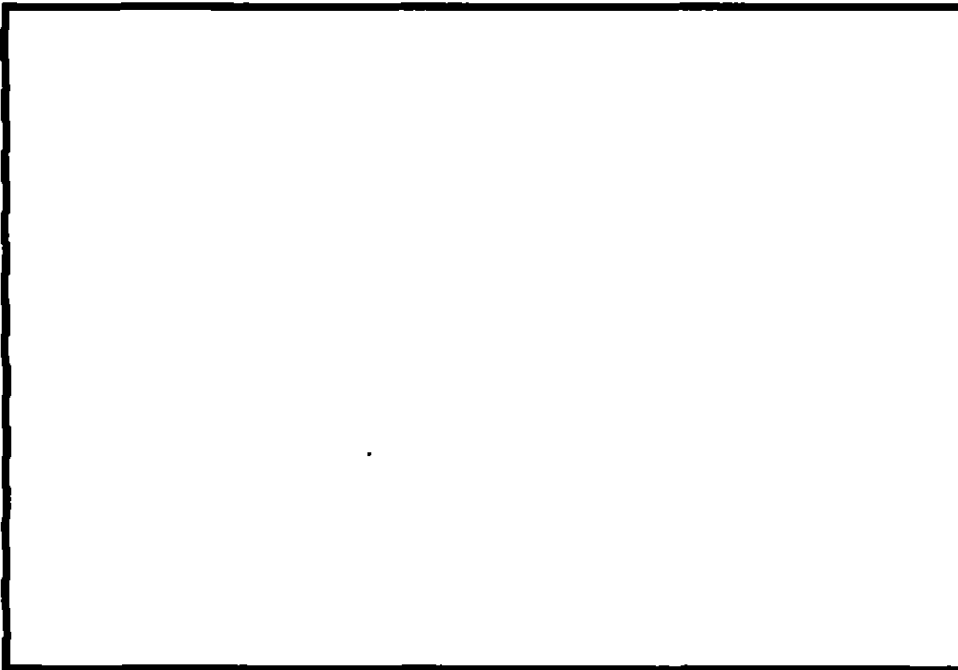
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ANEXOS

- ☒ Comprobante de pago de la tasa de presentación de la solicitud. \$443.000
- ☒ Documento que acredite la existencia y representación legal cuando el solicitante sea persona jurídica.
- ☒ Poderes, si fuere el caso. .Fotocopia Auténtica
- ☐ Documento de cesión del inventor al solicitante o a su causante.
- ☐ Declaraciones
- ☒ Copia de la solicitud internacional. (Resumen, descripción, reivindicaciones, dibujos)
- ☒ Traducción de la solicitud internacional al castellano. (Resumen, descripción, reivindicaciones, dibujos)
- ☐ Copia del examen preliminar efectuado por la IPEA.
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- ☒ Otros: Tarjeta de Propietarios

11 FIGURA CARACTERÍSTICA:



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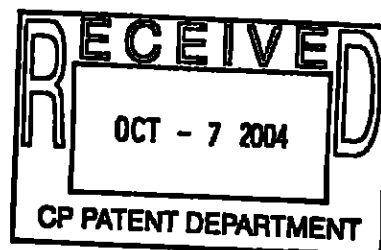
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(54) Title: LIQUID TOOTH WHITENING GEL.

(57) Abstract: A storage stable liquid peroxide whitening gel composition specified with an organic and inorganic particles.

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(54) Title: LIQUID TEXTILE WHITENING GEL.

(57) Abstract: A storage stable liquid peroxide whitening gel composition specified with an organic and inorganic particles.

LIQUID TOOTH WHITENING GEL**BACKGROUND OF THE INVENTION**

5

1. Field of the Invention

This invention relates in general to tooth whitening liquid gels and more particularly to a stable, aqueous, peroxide-containing liquid gel whitening product useful for whitening tooth enamel.

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2. The Prior Art

It has become desirable for a person's teeth to appear bright or "white". Society places a high value on the "whiteness" of one's teeth. One whose teeth are white may enjoy more personal confidence and satisfaction and may even enjoy greater social acceptance.

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A tooth is comprised of an inner dentin layer and an outer hard enamel layer that is the protective layer of the tooth. The enamel layer of a tooth is naturally an opaque white or slightly off-white ("ivory") color. It is the enamel and dentin layers that can become stained or discolored. The enamel layer of a tooth is composed of hydroxyapatite mineral crystals that create a somewhat porous surface. These hydroxyapatite crystals form microscopic hexagonal rods or prisms that make up the enamel surface. As a result, the surface of the enamel layer presents microscopic spaces or pores between the prisms. It is believed that this porous nature of the enamel layer is what allows staining agents and discoloring substances to permeate the enamel and discolor the tooth. These remaining substances can occupy the microscopic spaces and eventually alter the color of the tooth.

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Many substances that a person confronts or comes in contact with on a daily basis can "stain" or reduce the "whiteness" of one's teeth. In particular, the foods, tobacco products and fluids that one consumes tend to stain one's teeth. These products or substances tend to accumulate on the enamel layer of the tooth and form a pellicle film over the teeth. These staining and discoloring substances can then permeate the enamel layer. This problem occurs gradually over many years, but imparts a noticeable discoloration of the enamel of one's teeth. So long as the

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discolored teeth are still healthy and do not pose any health risk or problem, a product or substance that would whiten the discolored teeth would be advantageous.

5 It is also essential that a tooth whitening product that is to be used at home or in private by the consumer be safe and easy to use and be stable and retain its whitening efficacy during its storage on retail store shelves as well as over the period of use by the consumer.

10 Products and substances that are presently available to whiten teeth include a variety of different ingredients, but the primary active ingredient is a peroxide containing agent formulated into a liquid, solution, paste or gel. These products upon storage lose their whitening efficacy over time. A further limitation of commonly used aqueous peroxide solutions, is their brief period of efficacy when applied to the teeth in the oral cavity. For example, saliva, contains high concentrations of the enzyme catalase, which on contact, rapidly decomposes the peroxide into gaseous oxygen and water and so that there is only transitory
15 contact of the peroxide whitening agent with the teeth. In addition, the low viscosities of aqueous peroxide solutions do not allow the peroxide whitening agent to remain in contact with the teeth for as long as is necessary to effect substantive whitening because of the constant flushing effects of salivary secretions. This tendency toward rapid decomposition of peroxide and the rapid flushing away of the peroxide agent applied to the teeth has severely limited their
20 application to, and utility for, whitening teeth. It would be highly desirable, therefore, to provide a stable peroxide whitening liquid having increased retention on teeth to effect substantive whitening.

25 In copending US patent application Serial No. 10/155,496 filed May 24, 2002 there is disclosed a liquid gel dental whitening composition containing a peroxide whitening constituent dispersed in an aqueous liquid vehicle in which is dispersed a film forming component, the liquid gel rapidly drying when applied to the tooth surfaces to form in situ a gel-like film containing the peroxide whitening agent.

30 In one embodiment of the invention disclosed in USSN 10/155,496 there is provided an aqueous tooth whitening liquid gel having enhanced stability and whitening efficacy, the liquid gel being comprised of an aqueous vehicle containing a film forming combination of an

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ethylene oxide linear homopolymer, a Carbomer and a peroxide whitening agent, the pH of the composition being maintained at an acidic level.

5 The whitening liquid gel of USSN 10/155,496 is a portable oral care tooth whitener that can be conveniently painted onto the tooth surface. Upon the paint-on application to the teeth, the applied liquid whitening composition rapidly dries to produce, in situ, an adherent film of a thick gel that has the capacity to release the peroxide whitening agent over an extended period of time. The film adheres to the tooth surface whereby the released peroxide source then whitens the teeth to which the film is applied, the film being sufficiently adherent to counteract
10 the tooth flushing action of saliva generated in the oral cavity. The adjustment and maintenance of the composition pH to acid levels provides a peroxide source that is stable to decomposition on storage.

15 A problem encountered in the use of the liquid whitening gel of USSN 10/155,496 is that the composition is a clear, colorless product and the user when applying the product to tooth surfaces is uncertain, because of the product transparency, whether or not the tooth surfaces selected for whitening have had applied thereto the complete required application of the product. Less than complete application of the product to tooth surfaces can result in incomplete or reduced whitening results.

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Attempts to opacify the clear product with opaque pigments particles encounter the problem that such opacifiers when present in the liquid whitening gel have been found to be interactive with, and decompose, the peroxide ingredient with the result that the whitening efficacy of the gel is reduced, providing undesirable uneven tooth whitening.

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SUMMARY OF THE INVENTION

In accordance with the present invention there is provided a stable, opaque liquid whitening gel which can be visualized by the user after application to tooth surfaces which gel comprises a peroxide containing whitening agent dispersed in an orally acceptable vehicle comprising a film forming ingredient and opacifying particles selected from polymer particles, titanium oxide and titanium dioxide coated mica particles, the peroxide containing whitening agent being substantially stable in the presence of the opacifying particles.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The vehicle used to prepare the liquid gel whitening composition of the present invention preferably includes a non-toxic volatile alcohol or any suitable mixture thereof. The presence of the volatile alcohol imparts a rapid drying property to the applied liquid gel whitening composition and is present in the composition at a concentration of about 10 to about 50% by weight and preferably about 25 to about 40% by weight. A monohydric alcohol such as ethanol is preferred as a volatile solvent. Water is included in the vehicle of the composition and about 5 to about 30% by weight of the composition and preferably about 10 to about 20% by weight.

The proportion of vehicle used to prepare the liquid gel composition of the present invention is generally within the range of about 40 to about 80% by weight of the invention and preferably about 40 to about 60% by weight of the composition. A humectant such as sorbitol, glycerin, propylene glycol, PEG 600 is present in the vehicle of the present invention at a concentration of about 2 to about 15% by weight and preferably about 3 to about 8 by weight.

Examples of poly(ethylene oxides) useful in the practice of the present invention include PEG 2M, 5M, 7M, 14M, 23M, 45M and 90M commercially available from Union Carbide, Danbury, Connecticut, ranging in molecular weight from 100,000 to 4 million. A poly(ethylene oxide) preferred for use in the practice of the present invention is a poly(ethylene oxide) having a molecular weight of about 100,000. Such poly(ethylene oxide) or PEG 2M is a

nonionic polymer of ethylene oxide having an average molecular weight of 100,000 and has the general formula:



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wherein n represents the number of repeating $\text{CH}_2\text{CH}_2\text{O}$ groups.

Film forming agents useful in the practice of the present invention include Carbomers such as carboxymethylene polymers including acrylic acid polymers, and acrylic acid copolymers. Carboxypolymethylene is a slightly acidic vinyl polymer with active carboxyl groups. A
10 carboxypolymethylene preferred for use in the practice of the present invention is a water dispersible copolymer of acrylic acid cross-linked with approximately 0.75% to approximately 1.5% polyallyl sucrose that is sold under the trade designation Carbopol 934 or 974 by B.F. Goodrich. The Carbopol product is present in the liquid whitening composition of the present invention at a concentration of about 0.25 to about 1.5% by weight and preferably about 0.5 to
15 about 1.0% by weight.

Peroxide containing compounds which may be used as whitening agents in the practice of the present invention include hydrogen peroxide, urea peroxide and percarbonate salts such as sodium percarbonate persulfate salts such as sodium persulfate and perborate salts such as
20 sodium perborate. Most preferred is hydrogen peroxide. The peroxide containing compound is present in the liquid whitening gel compositions of the present invention at a concentration that will provide about 0.3 to about 12% by weight and preferably about 1 to about 10% by weight, active peroxide to the whitening gel formulation.

25 At the acid pH levels at which the composition of the present invention is maintained the Carbomer of the Carbopol type behaves like a liquid gel rather than a solid gel so that the Carbomer in combination with the poly(ethylene oxide) constituent provides thickness to the liquid product while maintaining a suitable consistency enabling the product to be painted on the tooth surface with a soft applicator brush.

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Polymeric opacifying particles useful in the practice of the present invention include olefin and fluoro polymers that may be incorporated in the liquid gel whitening compositions of the

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present invention which are unreactive with peroxide whitening agents present in the gel, and the gel once applied to the teeth forms a thin continuous film which can be visualized by the user, such polymers including polyethylene, polypropylene and ethylene/proylene copolymers, polytetrafluoro-ethylene and polyhexafluoropropene. The opacifying polymers are

5 incorporated in the liquid gel as powders having an average particle size in the range of about 0.5 to about 100 microns and preferably from about 1 micron to about 20 microns. Olefin polymers preferred in the practice of the present invention are polyethylene and polypropylene powders. Such powders are available from Micropowders, Inc. under the trade designation Micropoly 220 (polyethylene). Whitepoly 50. Microslip 519 (polytetrafluoroethylene).

10 The polymeric opacifying particles are incorporated in the liquid whitening gel compositions of the present invention at concentrations of about 1 to about 30% by weight and preferably about 3 to about 9% by weight without affecting the stability of the peroxide whitening agent present in the liquid whitening gel.

15 Titanium dioxide and titanium dioxide coated mica particles having an average particle size range of about 0.5 to about 75 microns and preferably about 5 to about 60 microns are also useful opacifying agents in liquid whitening gel of the present invention except that the concentration of these particles in the gel composition are used in the range of up to 1.0%, and preferably between 0.15% to 1.0% by weight. Higher concentration levels of the titanium
20 dioxide and titanium dioxide coated mica particles have tendency to destabilize the peroxide whitening agent during storage of the liquid whitening gel.

The liquid whitening gel composition of the present invention may also contain a flavoring agent. Flavoring agents that are used in the practice of the present invention include natural or
25 synthetic essential oils as well as various flavoring aldehydes, esters, alcohols, and similar materials. Examples of the essential oils include oils of spearmint, peppermint, wintergreen, sassafras, clove, sage, eucalyptus, marjoram, cinnamon, lemon, lime, grapefruit, and orange. Also useful are such chemicals as menthol, carvone, and anethole. Of these, the most commonly employed are the oils of peppermint, spearmint and wintergreen. The flavoring
30 agent is incorporated in the whitening liquid composition of the present invention at a concentration of about 0.1 to about 2% by weight and preferably about 0.1 to about 0.5% by weight.

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The liquid gel whitening composition of the present invention is initially prepared in the form of a liquid varnish and applied as such to the users teeth as by painting the teeth with a soft applicator. After application by the user, the alcohol and water vehicle constituents rapidly evaporate to leave a film or coating of a thick liquid gel on the teeth to which the varnish has been applied. The deposited film is comprised of the poly(ethylene oxide) constituent, the Carbomer constituent, the opacifier and the peroxide whitening agent or agents. The presence of the poly(ethylene oxide) and Carbomer constituent combination permits a slow release of the peroxide agent or agents to the applied tooth site, providing prolonged whitening treatment of the site.

The deposited film of liquid gel contains no ingredients imparting thereto an unacceptable taste or texture, rendering it unpleasant to the user and adheres strongly to tooth enamel. After application to the teeth, the composition dries in a relatively short time period, for example, 20 to 60 seconds to yield a strongly adherent, even layered opaque film which can be easily visualized by the user and distinguished from the tooth surface during application and while in place. The film gel is sufficiently strong and adherent enough to remain on the teeth for a period of time, for example up to 30 minutes to effect a whitening result and will resist the forces commonly applied by the lips and tongue. While the film is in place, the user is to refrain from mastication. The film can be removed as and when required, at will, by an employment of standard oral hygiene procedures such as brushing or by rinsing with an alcoholic mouthwash. While in place the film releases agents contained therein at a slow, relatively constant rate and in concentration sufficient effectively to effect stain removal from the teeth.

The whitening benefit imparted by the whitening gel of the present invention may be enhanced by brushing the teeth, before or after the application of the whitening gel, with a whitening toothpaste containing peroxide whitening agents or stain removing silica or alumina abrasives or a rinse composition having an alkaline pH as disclosed in US 6,174,516.

The liquid whitening gel compositions of the present invention are prepared by adding and mixing the ingredients of the composition in a suitable vessel such as a stainless steel tank

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provided with a mixer. In the preparation of the liquid whitening composition, the ingredients are advantageously added to the mixer in the following order: solvent film forming agent, humectant and peroxide compound and any desired flavoring. The ingredients are then mixed to form a homogeneous dispersion/solution. Thereafter an opacifier such as polyethylene, polytetrafluoroethylene or titanium dioxide coated mica particles are added to the solution. The present invention is illustrated by the following example but is not to be limited thereby.

Example

A liquid whitening gel base formula having a pH of 4.0 was prepared having the ingredients listed in Table I below:

TABLE I	
Liquid Whitening Gel Base Formula	
Ingredient	% by Weight
Water	10.07
Carbopol 974	1.00
95% Ethyl alcohol	34.8
Glycerin	5.00
PEG 600	10.00
PEG 2M	14.00
11hydrogen peroxide	25.00
85% Phosphoric acid	0.05
Monobasic sodium phosphate	0.05
Total	100.00

A series of liquid whitening gels was prepared in which the base formula of Table I was modified by either (1) 2 to 5% by weight a polyethylene (PE) powder having an average particle size of 6 to 8 microns; (2) 1 to 5% by weight of a polytetrafluoroethylene (PTFE) powder having particle size of 5 to 6 microns; (3) 0.8 to 2.5% by weight polypropylene powder having a particle size of 4 to 50 microns; (4) 2 to 5% by weight of the PE powder and 0.11 to 0.4% by weight of titanium dioxide powder having a particle size of 10 to 45 microns. The amounts of the polymer and inorganic titanium powders incorporated in the base formula are recorded in Tables II and III.

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TABLE II							
Liquid Whitening Gel Formula	A (Wt.%)	B (Wt.%)	C (Wt.%)	D (Wt.%)	E (Wt.%)	F (Wt.%)	G (Wt.%)
Base Formula	99.0	98.0	95.0	98.0	95.0	99.2	97.5
+ PE	--	--	--	2.0	5.0	--	--
+ PTFE	1.00	2.0	5.0	--	--	--	--
+ PP	--	--	--	--	--	0.8	2.5

TABLE III						
Liquid Whitening Gel Formula	GA (Wt.%)	H (Wt.%)	I (Wt.%)	J (Wt.%)	K (Wt.%)	L (Wt.%)
Base Formula	99.5	97.90	97.80	97.60	94.90	99.5
+ PE	--	2.00	2.00	2.00	5.00	--
+ TiO ₂	--	0.10	0.20	0.40	0.10	--
+ TiO ₂ coated mica	0.5	--	--	--	--	--

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The shelf stability of the liquid whitening gel formulations A-K, packaged in plastic bottles was determined by the percent of active oxygen present in the whitening liquid after a two week exposure of plastic bottle packaged whitening compositions to a temperature of 120°F (49°C).

- 5 Active oxygen analysis was performed using a standard iodometric titration method. The stability results are recorded in Table IV below.

TABLE IV				
Active Oxygen Analysis				
Liquid Whitening Gel Formula	Initial	Final	% Loss*	
A	9.07	8.95	None	
B	9.22	8.78	None	
C	8.79	8.28	5.9%	
D	8.83	9.08	None	
E	8.76	8.57	None	
F	9.01	8.88	None	
G	8.50	8.97	None	
GA	9.05	8.95	None	
H	8.97	9.13	None	
I	9.10	8.85	None	
J	8.63	8.70	None	
K	8.61	8.41	None	
L	9.05	8.95	None	

- 10 *Loss of <5% is considered experimental error.

The active oxygen analysis results recorded in Table IV indicate that the liquid whitening gel of the present invention retained sufficient peroxide content to be an efficacious whitening composition even after two (2) weeks of storage at the elevated temperature of 120°F (49°C).

- 15 Past experience with this temperature stability test indicated that when peroxide loss is less than 25% after two weeks at 120°F, a minimum shelf life of six (6) months can be expected.

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Example II

Following in the procedure of Example I, a liquid whitening gel designated "Gel L" was prepared having the ingredients listed in Table V in which 0.38% by weight of titanium dioxide particles having a particle size of 10 to 45 microns was incorporated. Gel L packaged in plastic bottles after two (2) weeks at 120°F active oxygen analysis indicated an oxygen loss of less than 5%.

TABLE V	
Liquid Gel Formula	Gel L (W%)
Carbomer	1.00
PEG 600	9.96
BHT	0.03
Glycerin	4.98
Water	17.00
Ethyl alcohol	34.67
PEG 2M	13.95
H2O2 (35%)	17.93
Sodium Phosphate	0.05
Phosphoric acid	0.05
Titanium dioxide	0.38

For purposes of comparison, the procedure of the Example II was repeated except the base formula was modified with 5% by weight TiO_2 designated "Gel M". After two (2) weeks at 120°F, active oxygen analysis indicated that Gel M had an unacceptable oxygen loss of 41.24%

For purposes of further comparison, a liquid whitening gel formula of the prior art (US 5,425,953) designated Gel N, the ingredients of which are listed in Table VI, below. Gel N was modified with 0.19 and 0.38% by weight of titanium oxide particles. These gels designated Liquid Gel Formula O and P were also prepared using the ingredients listed in Table VI below.

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TABLE VI		
Liquid Whitening Gel Formulation	N (Wt. %)	O (Wt. %)
Water	28.95	29.27
Ethanol	50.50	51.80
Hydroxypropyl cellulose	12.45	12.45
Carbamide peroxide	8.00	6.00
Calcium EDTA	0.10	0.10
Titanium dioxide	0.00	0.38

5 The shelf stability of whitening gels N and O packaged in plastic bottles was determined as the percent peroxide recovered from the gels after a 2 week exposure at 120°F (49°C). Percent peroxide recovery was performed using standard iodometric titration method. The stability results are recorded in Table VII below.

TABLE VII			
LIQUID WHITENING GEL STABILITY			
Liquid Whitening Gel Formula	Initial Wt. % H ₂ O ₂	Wt. % H ₂ O ₂ After 2 Weeks at 120°F	% Recovery of H ₂ O ₂
N	7.89	6.00	76
O	5.54	0.67	12

Claims

5 What is claimed is:

1. A stable opaque liquid tooth whitening gel composition suitable for application to teeth in the oral cavity comprising a peroxide containing compound dispersed in an orally acceptable vehicle containing a solvent, a film forming agent and an opacifier selected from organic and inorganic powders having a particle size of about 0.5 to 100 microns.
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2. The composition of claim 1 wherein the vehicle is comprised of water and a monohydric alcohol.
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3. The composition of claim 1 wherein the film forming agent is a mixture of a poly(ethylene) oxide and a Carbomer.
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4. The composition of claim 3 wherein the poly(ethylene oxide) is present in the composition in an amount from about 5 to about 50 weight %.
5. The composition of claim 3 wherein the Carbomer is a cross-linked acrylic acid copolymer.
6. The composition of claim 3 wherein the Carbomer is present in an amount from about 0.25 to about 1.5 weight %.
25
7. The composition of claim 1 wherein the peroxide containing compound is urea peroxide or hydrogen peroxide.
8. The composition of claim 1 wherein the peroxide containing compound is present in the composition at a concentration that will provide about 0.3 to about 12% by weight peroxide to the composition.
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9. The composition of claim 1 wherein the concentration of the peroxide containing compound is present in the composition at a concentration that will provide about 1 to about 10% by weight peroxide to the composition.
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10. The composition of claim 1 wherein the opacifier is an organic polymer having an average particle size in the range of 1 to about 100 microns.
- 5 11. The composition of claim 1 wherein the organic opacifier has a particle size of about 1 to about 20 microns.
12. The composition of claim 10 wherein the organic polymer is polyethylene.
- 10 13. The composition of claim 10 wherein the organic polymer is polypropylene.
14. The composition of claim 10 where the organic polymer is polytetrafluoroethylene.
- 15 15. The composition of claim 1 wherein the organic opacifier is present in the composition at a concentration of about 1 to about 10% by weight.
16. The composition of claim 1 wherein the inorganic opacifier has a particle size of about 5 to about 60 microns.
- 20 17. The composition of claim 1 wherein the inorganic opacifier is titanium dioxide or titanium dioxide coated mica.
18. The composition of claim 1 wherein the inorganic opacifier is present in the composition at a concentration of about 0.10% to about 1% by weight.
- 25 19. The method for whitening teeth which comprises:
- (a) preparing a liquid gel tooth whitening composition of claim 1;
- (b) painting the composition into contact with the teeth to be whitened;
- 30 (c) maintaining the composition in contact with the teeth to effect tooth whitening.

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(54) Título: GEL BLANQUEADOR DENTAL LIQUIDO

(57) Resumen: Una composición blanqueadora líquida de peróxido opacada con partículas orgánicas e inorgánicas estable en almacenamiento.

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GEL BLANQUEADOR DENTAL LÍQUIDO

ANTECEDENTES DE LA INVENCION

1. Campo de la Invención

Esta invención se relaciona, generalmente, a un gel líquido blanqueador y particularmente a un producto de gel líquido blanqueador que contiene peróxido estable acuoso, útil para blanquear el esmalte del diente.

2. El Arte Previo

Se ha hecho deseable que la dentadura de una persona aparezca reluciente o "blanca". La sociedad le da un alto valor a la "blancura" de nuestra dentadura. El que tenga dentadura blanca puede gozar de más satisfacción y confianza personal, aún puede gozar de mayor aceptación social.

Un diente esta compuesto por una capa de dentina interior y una capa exterior de esmalte duro que constituye la capa protectora del diente. La capa de esmalte de un diente es un blanco opaco naturalmente, o medianamente de un color no tan blanco ("marfil"). Son las capas de esmalte y dentina las que se pueden manchar o decolorar. La capa de esmalte del diente se compone de cristales minerales de hidroxiapatita que generan una superficie algo porosa. Estos cristales hidroxiapatita forman fibras o prismas hexagonales microscópicas que constituyen la superficie del esmalte. Como resultado, la superficie de la capa del esmalte presenta espacios microscópicos o poros entre las prismas. Se cree que esta naturaleza porosa de la capa de esmalte es lo que permite que los agentes

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que manchan y sustancias decolorantes penetren el esmalte y decoloren el diente. Estas sustancias residuales pueden llenar los espacios microscópicos y eventualmente alterar el color del diente.

Muchas sustancias que la persona confronta o entra en contacto diariamente pueden "manchar" o reducir la "blancura" de la dentadura. En particular: los alimentos, productos de tabaco y los fluidos que uno consume tienden a manchar nuestros dientes. Estos productos o sustancias tienden a acumularse en la capa de esmalte del diente y formar una película sobre la dentadura. Estas sustancias que manchan y decolorantes pueden penetrar en la capa de esmalte. Este problema ocurre gradualmente a lo largo de varios años, pero imparte una decoloración del esmalte de nuestros dientes. Mientras que los dientes decolorados se mantengan sanos y no presenten problemas o riesgos para la salud, un producto o sustancia que blanquee los dientes decolorados, sería de ventaja.

También es esencial que un producto blanqueador del diente que ha de ser utilizado en el hogar o en privado por el consumidor, sea seguro y de uso fácil, y que retenga la eficacia de su blanqueo durante el almacenamiento en el negocio vendedor, así como durante el tiempo de uso por el consumidor.

Los productos y sustancias disponibles en la actualidad para blanquear la dentadura incluyen una variedad de ingredientes diferentes, pero el ingrediente activo principal es un agente que contiene peróxido formulado como líquido, solución, pasta o gel. Al ser almacenados, estos productos pierden su eficacia para blanquear en el tiempo. Una limitación adicional de las soluciones de peróxido acuoso utilizados comúnmente, es

la breve duración de su eficacia cuando se aplican a la dentadura en la cavidad oral. Por ejemplo, la saliva contiene altas concentraciones de la enzima de catalasa, la cual al entrar en contacto, rápidamente descomponen el peróxido en oxígeno gaseoso y agua, de modo que el diente solo queda transitoriamente en contacto con el agente blanqueador de peróxido. Además, las bajas viscosidades de las soluciones de peróxido acuoso no permiten que el agente blanqueador de peróxido permanezca en contacto con los dientes por tiempo suficiente necesario para efectuar un blanqueamiento sustantivo, debido a los efectos de lavado constante de las secreciones salivares. Esta tendencia a la rápida descomposición del peróxido y el rápido lavado del agente de peróxido aplicado a la dentadura, ha limitado severamente la aplicación para el blanqueo de la dentadura y la utilidad para la misma. Sería altamente deseable, por lo tanto, el poder suministrar un líquido blanqueador de peróxido estable que tenga una retención aumentada en la dentadura para efectuar un blanqueamiento sustantivo.

En solicitud de Patente de los Estados Unidos de América pendientes de aprobación, Sería N° 10/155,496 presentada el 24 de Mayo de 2002, se divulga una composición en gel dental blanqueador líquido que contiene un constituyente blanqueador de peróxido dispersado en un vehículo líquido acuoso, en el cual está dispersado un componente que forma una película, el gel líquido se seca rápidamente cuando es aplicado a la superficie del diente para formar una película tipo gel in situ que contiene el agente blanqueador de peróxido.

En una realización de la invención revelada en USSN 10/155,496 se

proporciona un gel líquido blanqueador de dentadura acuoso, que tiene una estabilidad mejorada y eficacia de blanqueado, el gel líquido estando constituida por un vehículo acuoso que contiene una combinación que forma una película de un homopolímero lineal de óxido de etileno, un Carbomero y un agente blanqueador de peróxido, el pH del compuesto siendo mantenida a un nivel ácido.

El gel líquido blanqueador de USSN 10/155,496 es un blanqueador de cuidados orales portátil que se puede untar de manera conveniente en la superficie del diente. Al se aplicado a la superficie del diente, la composición blanqueadora líquida aplicada, se seca rápidamente in situ, una película adherente de gel espesa que tiene la capacidad de liberar el agente blanqueador de peróxido durante un período de tiempo extendido. La película se adhiere a la superficie del diente, por lo que la fuente de peróxido liberada, blanquea luego los dientes a los que se haya aplicado la película, y la película siendo suficientemente adhesiva para contrarrestar la acción lavadora de la saliva generada en la cavidad oral. El ajuste y mantenimiento del pH a los niveles ácidos de la composición, provee una fuente de peróxido que se mantiene estable contra la descomposición durante el almacenamiento.

Un problema que se presenta con el uso del gel líquido blanqueador del USSN 10/155,496 es que la composición es un producto transparente, incoloro y el usuario al aplicar el producto a la superficie del diente está inseguro, debido a la transparencia del producto, si se ha aplicado o no la cantidad requerida del producto a la superficie del diente seleccionado para blanquear. La insuficiente aplicación del producto a las superficies de los

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dientes puede resultar en una aplicación incompleta o resultados reducidos de blanqueado.

Los intentos de dar opacidad el producto transparente con partículas de pigmento opaco, se encuentra con el problema de que tales elementos para opacar cuando están presentes en el gel líquido blanqueador, interactúan y descomponen el ingrediente del peróxido, con el resultado de que la eficacia de blanqueado del gel es reducida, ocasionando un blanqueado irregular del diente de aspecto indeseable.

DESCRIPCIÓN ABREVIADA DE LA INVENCION

Según la presente invención, se suministra una gel blanqueador, líquido, estable, opaco que el usuario puede visualizar después de la aplicación a la superficie del diente, dicho gel comprende un peróxido que contiene de un agente blanqueador disperso en un vehículo oralmente aceptable que contiene un ingrediente que forma una película y partículas que opacan seleccionadas de partículas de polímero, óxido de titanio y partículas de mica recubiertas con dióxido de titanio, el peróxido que contiene agente blanqueador siendo sustancialmente estable en presencia de las partículas que opacan.

DESCRIPCIÓN DE LAS REALIZACIONES PREFERIDAS

El vehículo utilizado en la preparación de la composición del gel líquido blanqueador de la presente invención incluye preferiblemente un alcohol volátil, no tóxico o cualquier mezcla apropiada del mismo. La presencia del alcohol volátil imparte una propiedad de secado rápido a la composición blanqueadora de gel líquida y está presente en composición a una aproximadamente 10 en peso a aproximadamente 50% en peso y

preferiblemente aproximadamente 25 hasta aproximadamente 40% en peso. Un alcohol monohídrico, tal como el etanol, es preferido como solvente volátil. El agua se incluye en el vehículo de la composición y aproximadamente del 5 a aproximadamente 30% en peso de la composición y preferiblemente aproximadamente del 10 a aproximadamente 20% en peso.

La proporción del vehículo utilizado en la preparación de la composición de gel líquido de la presente invención, generalmente esta en el intervalo de aproximadamente 40 a aproximadamente 80 % en peso de la invención y preferiblemente aproximadamente 40 a 60% en peso de la composición. Un humectante como sorbitol, glicerina, propilenglicol, PEG 600 está presente en el vehículo de la presente invención, a una concentración de aproximadamente 2 a aproximadamente 15% en peso y preferiblemente de aproximadamente 3 a 8 en peso.

Ejemplos de poli(óxidos de etileno) utilizados en la práctica de la presente invención incluyen PEG 2M, 5M, 7M, 14M, 23M, 45M, y 90M comercialmente disponibles en Union Carbide, Danbury, Connecticut, con un intervalo de peso molecular desde 100,000 hasta 4 millones. Un poli(óxido de etileno) preferido para el uso en la práctica de la presente invención es un poli(óxido de polietileno) que contiene de un peso molecular de aproximadamente 100,000. Tal poli(óxido de etileno) o PEG 2M es un polímero no iónico de óxido de etileno que contiene de un peso molecular promedio de 100,000 y con la fórmula general:



en la cual n representa el número de grupos repetidos de $\text{CH}_2\text{CH}_2\text{O}$.

Los agentes que forman la película útiles en la práctica de la presente invención incluyen Carbomeros tales como polímeros de carboximetileno incluyendo polímero de ácido acrílico, y copolímeros de ácido acrílico. El Carboxipólimetileno es un polímero vinílico ligeramente ácido con grupos carboxílicos activos. Un carboxiplímetileno preferido para el uso en la práctica de la presente invención es un copolímero de ácido acrílico reticulado que se dispersa en el agua con aproximadamente 0,75% hasta aproximadamente 1,5% de polialil sacarosa polialil que se vende bajo la designación comercial Carbopol 934 ó 974 de B.F. Goodrich. El producto Carbopol está presente en la composición líquida blanqueadora de la presente invención a una concentración de aproximadamente 0,25% a aproximadamente 1,5% en peso y preferiblemente aproximadamente 0,5% hasta aproximadamente 1,0% en peso.

Compuestos que contienen de peróxido que se pueden usar como agentes blanqueadores en la práctica de la presente invención incluyen peróxido de hidrógeno, peróxido de urea y sales de percarbonato tales como sales de persulfato percarbonato de sodio tales como persulfato de sodio y sales de perborato como perborato de sódio. La preferida de todos es peróxido de hidrógeno. El compuesto que contiene de peróxido está presente en las composiciones de gel líquido blanqueador de la presente invención a una concentración que suministra a aproximadamente 0,3 hasta aproximadamente 12% en peso y preferiblemente aproximadamente 1 hasta aproximadamente el 10% en peso, de peróxido activo en la formulación del gel blanqueador.

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A los niveles de pH ácido en los cuales la composición de la presente invención se mantiene, el Carbomero del tipo Carbopol se comporta como un gel líquido, en lugar de un gel sólido, de modo que el Carbomero, en combinación con el constituyente de poli(óxido de etileno) le da espesor al producto líquido mientras mantiene una consistencia adecuada permitiendo que el producto pueda ser pintado en la superficie del diente con una pincel de aplicación suave.

Las partículas poliméricas para opacar útiles en la práctica de la presente invención incluyen polímeros de olefina y de flúor que pueden ser incorporados en las composiciones blanqueadores de gel líquido de la presente invención que no son reactivos con los agentes blanqueadores del peróxido presente en el gel, y el gel una vez aplicada a los dientes forma una película delgada continua que puede ser visualizada por el usuario, tales polímeros incluyen polietileno, polipropileno y copolímeros de etileno/propileno, politetrafluoro etileno y polihexafluoropropeno. Los polímeros que opacan son incorporados en el gel líquido como polvos que tienen un tamaño de partícula en el promedio de aproximadamente 0,5 hasta aproximadamente 100 micrones y preferiblemente desde aproximadamente 1 micrón hasta aproximadamente 20 micrones. Los polímeros de olefina preferidos en la práctica de la presente invención son polvos de polietilenos y polipropilenos. Tales polvos están disponibles en Micropowders, Inc. bajo el nombre comercial de Micropoly 220 (polietileno), Whitepoly 50, Microslip 519 (politetrafluoroetileno). Las partículas de polímero para opacar se incorporan en las composiciones de gel líquido blanqueador de la presente invención en concentraciones de

aproximadamente 1 a aproximadamente 30% en peso y preferiblemente de aproximadamente 3% a 9% en peso sin afectar la estabilidad del agente blanqueador de peróxido presente en el gel blanqueador líquido.

Las partículas de mica cubiertas con dióxido de titanio y dióxido de titanio que tienen un tamaño de partícula promedio en el intervalo de aproximadamente 0,5 hasta aproximadamente 75 micrones y preferiblemente aproximadamente 5 hasta aproximadamente 60 micrones, también son útiles como agentes que opacan el gel blanqueador líquido de la presente invención, excepto que la concentración de estas partículas en la composición del gel se utilizan en el intervalo de hasta 1,0%, y preferiblemente entre 0,15% hasta 1,0% en peso. Concentraciones más elevadas de dióxido del titanio y las partículas de mica recubiertas de dióxido del titanio tienen la tendencia a desestabilizar el agente blanqueador de peróxido durante el almacenamiento del gel líquido blanqueador.

La composición de gel líquido blanqueador de la presente invención también puede contener un agente saborizante. Los agentes saborizantes que se utilizan en la práctica de la presente invención incluyen aceites esenciales naturales o sintéticos, así como varios aldehídos, ésters, alcoholes saborizantes, y materiales similares. Ejemplos de los aceites saborizantes incluyen los aceites de menta verde, de menta pirola, sasafrás, clavo, salvia, eucaliptos, mejorana, canela, limón, lima, pomelo, y naranja. También son útiles tales químicos como mentol, carvone y anetola. De estos, los de uso más común son los aceites de menta verde y de pirola. El agente saborizante es incorporado a la composición líquida blanqueadora

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de la presente invención a una concentración de aproximadamente 0,1 hasta aproximadamente 2% en peso, y preferiblemente desde aproximadamente 0,1 hasta aproximadamente 0,5% en peso.

La composición de gel blanqueador líquido gel de la presente invención se prepara inicialmente en la forma de barniz líquido y se aplica como tal a los dientes del usuario, pintando los dientes con un pincel suave. Después de la aplicación por el usuario, los componentes del vehículo de alcohol y agua se evaporan rápidamente para dejar una película o recubrimiento de gel líquido grueso en los dientes a los cuales el barniz se ha aplicado. La película depositada se compone de un constituyente de poli(óxido de etileno), el constituyente Carbomero, el opacador y agente o agentes blanqueadores de peróxido. La presencia de poli(óxido de etileno) y el constituyente de Carcomero, en combinación permite una liberación lenta del agente o agentes de peróxido al sitio de aplicación del diente, proporcionando un tratamiento de blanqueado prolongado en el sitio.

La película de gel líquido depositada no contiene ingredientes que impartan sabor o textura inaceptables, lo que sería desagradable para el usuario con adhesión fuerte al esmalte del diente. Luego de la aplicación a los dientes la composición se seca en un tiempo relativamente corto, por ejemplo, 20 a 60 segundos, para obtener una fuerte adhesión de película opaca de extensión uniforme que se puede visualizar fácilmente, y que se puede distinguir de la superficie del diente durante la aplicación y mientras este en posición. La película del gel es suficiente fuerte y de adherencia suficiente para permanecer en los dientes durante un tiempo, por ejemplo hasta por 30 minutos para realizar un resultado blanqueador y que resistirá

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las fuerzas comúnmente ejercidas por los labios y la lengua. Mientras la película permanece en posición, el usuario debe evitar masticar. La película se puede retirar cuando se requiera, a voluntad, mediante el empleo de procedimientos normales estándares de higiene oral, tales como el cepillado o con enjuague bucal alcoholizado. Mientras permanezca en posición, la película libera agentes contenidos en el mismo, a una lenta velocidad relativamente constante y en concentración suficiente efectiva para realizar la remoción de manchas de los dientes.

El beneficio blanqueador impartido por el gel blanqueador de la presente invención puede ser mejorada cepillandose los dientes, antes o después de la aplicación del gel blanqueador, con una pasta de dientes que contiene agentes blanqueadores de peróxido o silicio para la remoción de manchas o abrasivos de alumina o una composición de enjuague que tiene un pH alcalino como se divulga en US 6,174,516.

Las composiciones de gel blanqueador líquido de la presente invención son preparados mediante la adición y mezcla de ingredientes de la composición en un recipiente adecuado, tal como un tanque de acero inoxidable provisto con una mezcladora. En la preparación de la composición líquida blanqueadora, los ingredientes, se adicionan con ventaja a la mezcladora en el siguiente orden: agentes solventes de formación de película, humectante y compuesto de peróxido y cualquier saborizante deseado. Los ingredientes son entonces mezclados para formar un dispersión/solución homogénea. Después se adiciona un elemento para opacar, tal como polietileno, politetrafluoroetileno o dióxido de titanio o partículas de mica recubiertas con dióxido de titanio a la solución. La

presente invención se ilustra con el siguiente ejemplo, pero sin ser considerado limitativo de la misma.

Ejemplo I

Una fórmula en base a un gel blanqueador líquido con un pH de 4,0 se preparó con los ingredientes enumerados en la Tabla 1 que sigue:

TABLA 1	
Fórmula con Base de un Gel Blaueador Líquido	
Ingrediente	% por peso
Agua	10,07
Carbopol 974	1,00
Alcohol etílico al 95%	34,8
Glicerina	5,00
PEG 600	10,00
PEG 2M	14,00
Peróxido de hidrógeno	25,00
Ácido Fosfórico al 85%	0,05
fosfatato monobásico de sodio	0,05
Total	100.00

Una serie de geles líquidos blanqueadores se preparon en la cual la fórmula de base de la Tabla 1 se modificó por ya sea: (1) 2 al 5% por peso un polvo de polietileno (PE) que tiene un tamaño de partícula promedio de 6 a 8 micrones; (2) 1 a 5% en peso de un polvo de politetrafluoroetileno (PTFE) que tiene un tamaño de partículas de 5 a 6 micrones; (3) 0,8 a 2,5% en peso de polvo de polipropileno que tiene un tamaño de partícula de 4 a 50 micrones; (4) 2 a 5% en peso de polvo de PE y 0,11 a 0,4% en peso de polvo de dióxido de titanio que tiene un tamaño de partícula de 10 a 45 micrones. Las cantidades de polvos de polímero y titanio inorgánico,

incorporados en la formula base están registradas en las Tablas II y III.

TABLA II							
Fórmula de Gel Blanqueador Líquido	A (P%)	B (P%)	C (P%)	D (P%)	E (P%)	F (P%)	G (P%)
Fórmula de Base	99,0	98,0	95,0	98,0	95,0	99,2	97,5
+PE	--	--	--	2,0	5,0	--	--
+PTFE	1,00	2,0	5,0	--	--	--	--
+PP	--	--	--	--	--	0,8	2,5

TABLA III						
Fórmula de Gel Blanqueadora Líquido	GA (P%)	H (P%)	I (P%)	J (P%)	K (P%)	L (P%)
Fórmula de Base	99,5	97,90	97,80	97,60	94,90	99,5
+PE	--	2,00	2,00	2,00	5,00	--
+ TiO ₂	--	0,10	0,20	0,40	0,10	--
+ TiO ₂ mica recubierta	0,5	--	--	--	--	--

El tiempo de estabilidad imperecedero de las formulaciones A-K, empaquetadas en botellas de plástico se determinó por el porcentaje de oxígeno activo presente en el líquido blanqueador después de dos semanas de exposición en frascos plásticos de la composición blanqueadora empaquetada a un temperatura de 120°F (49°C). Se realizó un análisis de oxígeno activo utilizando el método estandar de titulación iodométrica. Los resultados de estabilidad se registraron en la Tabla IV a continuación.

TABLA IV Análisis de oxígeno activo			
Fórmula de Gel Blanqueador Líquido	Inicial	Final	% Pérdida*
A	9.07	8.95	Ninguna
B	9.22	8.78	Ninguna
C	8.79	8.28	5.9%
D	8.83	9.08	Ninguna
E	8.76	8.57	Ninguna
F	9.01	8.88	Ninguna
G	8.50	8.97	Ninguna
GA	9.05	8.95	Ninguna
H	8.97	9.13	Ninguna
I	9.10	8.85	Ninguna
J	8.63	8.70	Ninguna
K	8.61	8.41	Ninguna
L	9.05	8.95	Ninguna

*Pérdida <5% se considera error experimental

Los resultados del análisis de oxígeno activo registrados en la Tabla IV son indicativos de que el gel blanqueador líquido de la presente invención retuvo suficiente contenido de peróxido para ser una composición blanqueadora eficaz aún después de dos (2) semanas de almacenamiento a temperaturas elevadas de 120°F (49°C). Experiencias previas con esta prueba de estabilidad de temperatura indicaron que cuando la pérdida de peróxido es menor que 25% después de dos semanas a 120°F, se puede esperar un tiempo de estabilidad imperecedero de seis (6) meses.

Ejemplo II

Siguiendo el procedimiento del Ejemplo I, se preparó un gel blanqueador líquido designado "Gel L" con los ingredientes enumerados en la Tabla V en la cual se incorporaron 0.35% en peso de partículas de

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dióxido de titanio que tienen un tamaño de partícula de 10 a 45 micrones. Gel L, empaquetada en envases de plástico, después de dos (2) semanas a temperatura de 120°F, el análisis de oxígeno activo indicó una pérdida de oxígeno menor del 5%.

TABLA V	
Fórmula de Gel Líquida	Gel L (W%)
Carbomero	1,00
PEG 600	9,96
BHT	0,03
Glicerina	4,98
Agua	17,00
Alcohol Etílico	34,67
PEG 2M	13,95
H2O2 (35%)	17,93
Fosfato de Sodio	0,05
Ácido Fosfórico	0,05
Dióxido de titanio	0,38

Con propósitos de comparación, el procedimiento del Ejemplo II se repitió, salvo que la fórmula base se modificó con 5% en peso de TiO_2 designado "Gel M". Después de dos semanas a una temperatura de 120°F, el análisis del oxígeno activo dio una indicación que el Gel M mostraba una pérdida de oxígeno inaceptable del 41,24%.

Con propósitos de comparación adicional, una fórmula de gel blanqueador líquido del arte previo (US 5,425,953) designada Gel N, cuyos ingredientes se enumeran en la Tabla VI, en adelante. Gel N se modificó con 0,19 y 0,38% en peso de partículas de óxido de titanio. Estos geles designadas Fórmula Gel Líquida O y P se preparó también utilizando los ingredientes enumerados en la Tabla VI que sigue.

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TABLA VI		
Formulación de Gel Blanqueador Líquido	N(P%)	O(P%)
Agua	28,95	29,27
Etanol	50,50	51,80
Hidroxipropil celuloso	12,45	12,45
Peróxido de Carbamida	8,00	6,00
Cálcio EDTA	0,10	0,10
Dióxido de Titanio	0,00	0,38

La estabilidad de almacenamiento de los geles blanqueadores N y O, empaquetados en frascos plásticos se determinó como el porcentaje de peróxido recuperado de los geles después de 2 semanas de exposición a 120°F (49°C). La recuperación del peróxido se realizó utilizando el método estandar de titulación iodométrica. Los resultados de estabilidad se registraron en la Tabla VII que sigue.

TABLA VII ESTABILIDAD DEL GEL BLANQUEADOR LÍQUIDO			
Fórmula de Gel Blanqueador Líquido	P%H ₂ O ₂ Inicial	P%H ₂ O ₂ después de 2 semanas a 120° F	% Recup. de H ₂ O ₂
N	7,89	6,00	76
O	5,54	0,67	12

REIVINDICACIONES

1. Un compuesto de gel líquido, estable, opaco para blanquear dentadura adecuada para su aplicación a los dientes de la cavidad oral que consiste en un compuesto que contiene peróxido para esparcir de manera oralmente aceptable caracterizado porque contiene un solvente, un agente formador de una película y opacador seleccionado de polvos orgánicos e inorgánicos que tiene partículas de tamaño aproximadamente del 0,5 a 100 micrones.
2. Una composición de la reivindicación 1 caracterizada porque el agente formador de película esta constituido por agua y un alcohol monohídrico.
3. Una composición de la reivindicación 1 caracterizada porque el vehículo está constituido por una mezcla de óxido de poli(etileno) y un Carbomero.
4. Una composición de la reivindicación 3 caracterizada porque el óxido de poli(etileno) esta presente en el compuesto en una cantidad desde aproximadamente 5 hasta aproximadamente 50 % en peso.
5. Una composición de la reivindicación 3 caracterizada porque el Carbomero es un copolímero de ácido acrílico reticulado.
6. Una composición de la reivindicación 3 caracterizada porque el Carbomero esta presente en una cantidad desde aproximadamente 0,25 hasta aproximadamente 1,5 % en peso.
7. Una composición de la reivindicación 1 caracterizada porque el compuesto que contiene peróxido es un peróxido de urea o un peróxido de hidrógeno.

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8. Una composición de la reivindicación 3 caracterizada porque el compuesto que contiene peróxido esta presente en la composición en una concentración que proveerá aproximadamente 0,3 hasta aproximadamente 12% en peso de peróxido al compuesto.
9. Una composición de la reivindicación 3 caracterizada porque la concentración del compuesto que contiene peróxido esta presente en el compuesto a una concentración que proveerá aproximadamente 1 hasta aproximadamente 10% en peso de peróxido al compuesto.
10. La composición de la reivindicación 1, caracterizada porque el elemento para opacar es un polímero orgánico que tiene un tamaño de partícula promedio en el intervalo de 1 a aproximadamente 100 micrones.
11. La composición de la reivindicación 1, caracterizada porque el elemento para opacar orgánico tiene un tamaño de partícula de aproximadamente 1 a aproximadamente 20 micrones.
12. La composición de la reivindicación 10, caracterizada porque el polímero orgánico es polietileno.
13. La composición de la reivindicación 10, caracterizada porque el polímero orgánico es polipropileno.
14. La composición de la reivindicación 10, caracterizada porque el polímero orgánico es politetrafluoroetileno.
15. La composición de la reivindicación 1, caracterizada porque el elemento para opacar orgánico está presente en la composición a una concentración de aproximadamente 1 a aproximadamente 10% en peso.
16. La composición de la reivindicación 1, caracterizada porque el elemento para opacar tiene un tamaño de partícula de aproximadamente 5 a

aproximadamente 60 micrones.

17. La composición de la reivindicación 1, caracterizada porque el elemento para opacar inorgánico es dióxido de titanio o mica recubierta con dióxido de titanio.

18. La composición de la reivindicación 1, caracterizada porque el elemento para opacar inorgánico está presente en la composición a una concentración de aproximadamente 0,10% a aproximadamente 1% en peso.

19. El método para blanquear dientes que comprende:

(a) preparar una composición en gel blanqueadora dental líquida de la reivindicación 1;

(b) pintar la composición en contacto con los dientes a blanquear;

(c) mantener la composición en contacto con los dientes para efectuar el blanqueado de los dientes.

BUENOS AIRES, ENERO DE 2004

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RESUMEN

GEL BLANQUEADOR DENTAL LÍQUIDO

Un compuesto de gel blanqueador líquido con peróxido estable durante el almacenamiento opaco con unas partículas orgánicas e inorgánicas.

TRANSMITTAL LETTER TO THE
UNITED STATES RECEIVING OFFICE

Date	15 January 2004
International Application No.	
Attorney Docket No.	IR 7001-00

I. Certification under 37 CFR 1.10 (If applicable)

EV 380773855 US
Express Mail mailing number

15 January 2004
Date of Deposit

I hereby certify that the application/correspondence attached hereto is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to Assistant Commissioner for Patents, Washington, D.C. 20231.

Carol Stanley
Signature of person mailing correspondence

Carol Stanley
Typed or printed name of person mailing correspondence

II. ☒ New International Application

TITLE	LIQUID TOOTH WHITENING GEL
-------	----------------------------

Earliest priority date (Day/Month/Year)
15 January 2003

SCREENING DISCLOSURE INFORMATION: In order to assist in screening the accompanying international application for purposes of determining whether a license for foreign transmittal should and could be granted and for other purposes, the following information is supplied. (Note: check as many boxes as apply):

- A. ☐ The invention disclosed was not made in the United States.
- B. ☐ There is no prior U.S. application relating to this invention.
- C. ☒ The following prior U.S. application(s) contain subject matter which is related to the invention disclosed in the attached international application. (NOTE: priority to these applications may or may not be claimed on form PCT/RO/101 (Request) and this listing does not constitute a claim for priority).

application no.	USSN 10/342,078	filed on	15 January 2003
application no.		filed on	

- D. ☐ The present international application ☐ is identical ☐ contains less subject matter than that found in the prior U.S. application(s) identified in paragraph C.
- E. ☐ The present international application ☐ contains additional subject matter not found in the prior U.S. application(s) identified in paragraph C. above. The additional subject matter is found on pages and ☐ DOES NOT ALTER ☐ MIGHT BE CONSIDERED TO ALTER the general nature of the invention in a manner which would require the U.S. application to have been made available for inspection by the appropriate defense agencies under 35 U.S.C. 181 and 37 CFR 5.1. See 37 CFR 5.15

III. ☐ A Response to an Invitation from the RO/US. The following document(s) is (are) enclosed:

- A. ☐ A Request for An Extension of Time to File a Response
- B. ☐ A Power of Attorney (General or Regular)
- C. ☐ Replacement pages:

pages		of the request (PCT/RO/101)	pages		of the figures
pages		of the description	pages		of the abstract
pages		of the claims			

- D. ☐ Submission of Priority Documents

Priority document		Priority document	
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- E. ☐ Fees as specified on attached Fee Calculation sheet form PCT/RO/101 annex

IV. ☐ A Request for Rectification under PCT 91 ☐ A Petition ☐ A Sequence Listing DisketteV. ☐ Other (please specify):

The person signing this form is the:	☐ Applicant	Michael J. McGreal
	☒ Attorney Agent (Reg. No.) 25 398	Typed name of signor
	☐ Common Representative	<i>Michael J. McGreal</i> Signature

S0556E2209EVA3

PCT REQUEST

Original (for SUBMISSION)

0	For receiving Office use only	
0-1	International Application No.	
0-2	International Filing Date	
0-3	Name of receiving Office and "PCT International Application"	
0-4	Form - PCT/RO101 PCT Request	
0-4-1	Prepared Using	PCT-SAFE [EASY mode] Version 3.50 (Build 0002.153)
0-5	Petition The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty	
0-6	Receiving Office (specified by the applicant)	United States Patent and Trademark Office (USPTO) (RO/US)
0-7	Applicant's or agent's file reference	7091-00
I	Title of Invention	LIQUID TOOTH WHITENING GEL
II	Applicant	
II-1	This person is:	applicant only
II-2	Applicant for	all designated States
II-4	Name:	COLGATE-PALMOLIVE COMPANY
II-5	Address:	300 Park Avenue New York, New York 10022 United States of America
II-6	State of nationality	US
II-7	State of residence	US
II-8	Telephone No.	732-878-7157
II-9	Facsimile No.	732-878-7853
II-10	e-mail	allen_park@colpal.com
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III-1-4	Name (LAST, First)	HOIC, Diego, A.
III-1-5	Address:	153 Walnut Court Highland Park, New Jersey 08904 United States of America

PCT REQUEST

Original (for SUBMISSION)

III-2 III-2-1 III-2-4 III-2-5	Applicant and/or inventor This person is: Name (LAST, First) Address:	inventor only SABELKO, Jobiah 27D Chicopee Drive Princeton, New Jersey 08540 United States of America
III-3 III-3-1 III-3-4 III-3-5	Applicant and/or inventor This person is: Name (LAST, First) Address:	inventor only MANDADI, Prakasarao 29 Crestmont Drive Hillsborough, New Jersey 08844 United States of America
III-4 III-4-1 III-4-4 III-4-5	Applicant and/or inventor This person is: Name (LAST, First) Address:	inventor only PATEL, Suryakant 103 Pine Street Bridgewater, New Jersey 08807 United States of America
III-5 III-5-1 III-5-4 III-5-5	Applicant and/or inventor This person is: Name (LAST, First) Address:	inventor only DIXIT, Nagara 3 Titus Lane Plainsboro, New Jersey 08536 United States of America
III-6 III-6-1 III-6-4 III-6-5	Applicant and/or inventor This person is: Name (LAST, First) Address:	inventor only PRENCIPE, Michael 39 Spruce Street West Windsor, New Jersey 08550 United States of America

PCT REQUEST

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IV-1	Agent or common representative; or address for correspondence The person identified below is hereby/ has been appointed to act on behalf of the applicant(s) before the competent International Authorities as:	agent
IV-1-1	Name (LAST, First)	PARK, Ellen, K.
IV-1-2	Address:	Colgate-Palmolive Company 909 River Road P.O. Box 1343 Piscataway, New Jersey 08855 United States of America
IV-1-3	Telephone No.	732-878-7157
IV-1-4	Facsimile No.	732-878-7853
IV-1-5	e-mail	ellen_park@colpal.com
IV-1-6	Agent's registration No.	34055
IV-2	Additional agent(s)	additional agent(s) with same address as first named agent
IV-2-1	Names(s)	MC GREAL, Michael, J. (25,356)
V	DESIGNATIONS	
V-1	The filing of this request constitutes under Rule 4.3(a), the designation of all Contracting States bound by the PCT on the international filing date, for the grant of every kind of protection available and, where applicable, for the grant of both regional and national patents.	
VI-1	Priority claim of earlier national application	
VI-1-1	Filing date	15 January 2003 (15.01.2003)
VI-1-2	Number	10/342,076
VI-1-3	Country	US
VI-2	Priority document request The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) identified above as item(s):	VI-1
VII-1	International Searching Authority Chosen	European Patent Office (EPO) (ISA/EP)

PCT REQUEST

Original (for SUBMISSION)

VII	Declarations	Number of declarations	
VII-1	Declaration as to the identity of the inventor	-	
VII-2	Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent	1	
VII-3	Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application	1	
VII-4	Declaration of inventorship (only for the purposes of the designation of the United States of America)	-	
VII-5	Declaration as to non-prejudicial disclosures or exceptions to lack of novelty	-	

PCT REQUEST

Original (for SUBMISSION)

VII-2-1	Declaration: Entitlement to apply for and be granted a patent Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent (Rules 4.17(ii) and 51bis.1(a)(ii)). In a case where the declaration under Rule 4.17(iv) is not appropriate: Name (LAST, First)	in relation to this international application COLGATE-PALMOLIVE COMPANY is entitled to apply for and be granted a patent by virtue of the following:
VIII-2-1(iv)		an assignment from HOIC Diego, A. to COLGATE-PALMOLIVE COMPANY, dated 10 January 2003 (10.01.2003)
VIII-2-1(iv)		an assignment from SABELKO Jobiah to COLGATE-PALMOLIVE COMPANY, dated 14 January 2003 (14.01.2003)
VIII-2-1(iv)		an assignment from MANDADI Prakasarao to COLGATE-PALMOLIVE COMPANY, dated 13 January 2003 (13.01.2003)
VIII-2-1(iv)		an assignment from PATEL Suryakant to COLGATE-PALMOLIVE COMPANY, dated 13 January 2003 (13.01.2003)
VIII-2-1(iv)		an assignment from DIXIT Nagaraj to COLGATE-PALMOLIVE COMPANY, dated 10 January 2003 (10.01.2003)
VIII-2-1(iv)		an assignment from PRENCIPE Michael to COLGATE-PALMOLIVE COMPANY, dated 13 January 2003 (13.01.2003)
VIII-2-1(ix)	This declaration is made for the purposes of:	all designations

PCT REQUEST

Original (for SUBMISSION)

VIII-3-1	Declaration: Entitlement to claim priority Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application specified below, where the applicant is not the applicant who filed the earlier application or where the applicant's name has changed since the filing of the earlier application (Rules 4.17(ii) and 51bis.1(a)(iii)) Name:	in relation to this international application COLGATE-PALMOLIVE COMPANY is entitled to claim priority of earlier application No. 10/342,076 by virtue of the following:
VIII-3-1(iv)		an assignment from HOIC Diego, A. to COLGATE-PALMOLIVE COMPANY, dated 10 January 2003 (10.01.2003)
VIII-3-1(iv)		an assignment from SABELKO Jobiah to COLGATE-PALMOLIVE COMPANY, dated 14 January 2003 (14.01.2003)
VIII-3-1(iv)		an assignment from MANDADI Prakas Rao to COLGATE-PALMOLIVE COMPANY, dated 13 January 2003 (13.01.2003)
VIII-3-1(iv)		an assignment from PATEL Suryakant to COLGATE-PALMOLIVE COMPANY, dated 13 January 2003 (13.01.2003)
VIII-3-1(iv)		an assignment from DIXIT Nagaraj to COLGATE-PALMOLIVE COMPANY, dated 10 January 2003 (10.01.2003)
VIII-3-1(iv)		an assignment from PRENCIPE Michael to COLGATE-PALMOLIVE COMPANY, dated 13 January 2003 (13.01.2003)
VIII-3-1(iv)	(This declaration is made for the purposes of:	all designations

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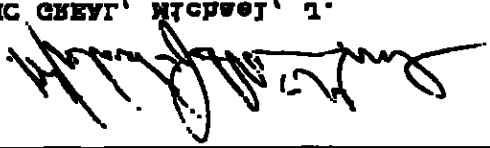
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10-1	substance information regarding date of actual receipt of the	

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X-1-1	Name:		
X-1	common information signature of official agent of		
IX-30	signature of agent of the information	EXDITP	
IX-10	document the parties; figure of the discovery which applied		
IX-11	PCT-STATE balance made	-	1
IX-8	for completion of the accomplishing item	-	-
IX-1	TOTAL	33	
IX-2	discovery	0	-
IX-4	approval	1	1
IX-3	discovery	3	-
IX-5	discovery	13	-
IX-1	signature (including completion of the)	1	-
IX	check list	number of pages	signature (if) attached

original (or submission)

PCT REQUEST

111

1081-00

LIQUID TOOTH WHITENING GEL**BACKGROUND OF THE INVENTION**

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1. Field of the Invention

This invention relates in general to tooth whitening liquid gels and more particularly to a stable, aqueous, peroxide-containing liquid gel whitening product useful for whitening tooth enamel.

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2. The Prior Art

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It has become desirable for a person's teeth to appear bright or "white". Society places a high value on the "whiteness" of one's teeth. One whose teeth are white may enjoy more personal confidence and satisfaction and may even enjoy greater social acceptance.

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A tooth is comprised of an inner dentin layer and an outer hard enamel layer that is the protective layer of the tooth. The enamel layer of a tooth is naturally an opaque white or slightly off-white ("ivory") color. It is the enamel and dentin layers that can become stained or discolored. The enamel layer of a tooth is composed of hydroxyapatite mineral crystals that create a somewhat porous surface. These hydroxyapatite crystals form microscopic hexagonal rods or prisms that make up the enamel surface. As a result, the surface of the enamel layer presents microscopic spaces or pores between the prisms. It is believed that this porous nature of the enamel layer is what allows staining agents and discoloring substances to permeate the enamel and discolor the tooth. These remaining substances can occupy the microscopic spaces and eventually alter the color of the tooth.

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Many substances that a person confronts or comes in contact with on a daily basis can "stain" or reduce the "whiteness" of one's teeth. In particular, the foods, tobacco products and fluids that one consumes tend to stain one's teeth. These products or substances tend to accumulate on the enamel layer of the tooth and form a pellicle film over the teeth. These staining and discoloring substances can then permeate the enamel layer. This problem occurs gradually over many years, but imparts a noticeable discoloration of the enamel of one's teeth. So long as the

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discolored teeth are still healthy and do not pose any health risk or problem, a product or substance that would whiten the discolored teeth would be advantageous.

It is also essential that a tooth whitening product that is to be used at home or in private by the consumer be safe and easy to use and be stable and retain its whitening efficacy during its storage on retail store shelves as well as over the period of use by the consumer.

Products and substances that are presently available to whiten teeth include a variety of different ingredients. but the primary active ingredient is a peroxide containing agent formulated into a liquid, solution, paste or gel. These products upon storage lose their whitening efficacy over time. A further limitation of commonly used aqueous peroxide solutions, is their brief period of efficacy when applied to the teeth in the oral cavity. For example, saliva, contains high concentrations of the enzyme catalase, which on contact, rapidly decomposes the peroxide into gaseous oxygen and water and so that there is only transitory contact of the peroxide whitening agent with the teeth. In addition, the low viscosities of aqueous peroxide solutions do not allow the peroxide whitening agent to remain in contact with the teeth for as long as is necessary to effect substantive whitening because of the constant flushing effects of salivary secretions. This tendency toward rapid decomposition of peroxide and the rapid flushing away of the peroxide agent applied to the teeth has severely limited their application to, and utility for, whitening teeth. It would be highly desirable, therefore, to provide a stable peroxide whitening liquid having increased retention on teeth to effect substantive whitening.

In copending US patent application Serial No. 10/155,496 filed May 24, 2002 there is disclosed a liquid gel dental whitening composition containing a peroxide whitening constituent dispersed in an aqueous liquid vehicle in which is dispersed a film forming component, the liquid gel rapidly drying when applied to the tooth surfaces to form in situ a gel-like film containing the peroxide whitening agent.

In one embodiment of the invention disclosed in USSN 10/155,496 there is provided an aqueous tooth whitening liquid gel having enhanced stability and whitening efficacy, the liquid gel being comprised of an aqueous vehicle containing a film forming combination of an

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ethylene oxide linear homopolymer, a Carbomer and a peroxide whitening agent. the pH of the composition being maintained at an acidic level.

5 The whitening liquid gel of USSN 10/155.496 is a portable oral care tooth whitener that can be conveniently painted onto the tooth surface. Upon the paint-on application to the teeth, the applied liquid whitening composition rapidly dries to produce, in situ, an adherent film of a thick gel that has the capacity to release the peroxide whitening agent over an extended period of time. The film adheres to the tooth surface whereby the released peroxide source then whitens the teeth to which the film is applied. the film being sufficiently adherent to counteract
10 the tooth flushing action of saliva generated in the oral cavity. The adjustment and maintenance of the composition pH to acid levels provides a peroxide source that is stable to decomposition on storage.

15 A problem encountered in the use of the liquid whitening gel of USSN 10/155.496 is that the composition is a clear, colorless product and the user when applying the product to tooth surfaces is uncertain, because of the product transparency, whether or not the tooth surfaces selected for whitening have had applied thereto the complete required application of the product. Less than complete application of the product to tooth surfaces can result in incomplete or reduced whitening results.

20 Attempts to opacify the clear product with opaque pigments particles encounter the problem that such opacifiers when present in the liquid whitening gel have been found to be interactive with, and decompose, the peroxide ingredient with the result that the whitening efficacy of the gel is reduced, providing undesirable uneven tooth whitening.

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SUMMARY OF THE INVENTION

In accordance with the present invention there is provided a stable, opaque liquid whitening gel which can be visualized by the user after application to tooth surfaces which gel comprises a peroxide containing whitening agent dispersed in an orally acceptable vehicle comprising a film forming ingredient and opacifying particles selected from polymer particles, titanium oxide and titanium dioxide coated mica particles. the peroxide containing whitening agent being substantially stable in the presence of the opacifying particles.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The vehicle used to prepare the liquid gel whitening composition of the present invention preferably includes a non-toxic volatile alcohol or any suitable mixture thereof. The presence of the volatile alcohol imparts a rapid drying property to the applied liquid gel whitening composition and is present in the composition at a concentration of about 10 to about 50% by weight and preferably about 25 to about 40% by weight. A monohydric alcohol such as ethanol is preferred as a volatile solvent. Water is included in the vehicle of the composition and about 5 to about 30% by weight of the composition and preferably about 10 to about 20% by weight.

The proportion of vehicle used to prepare the liquid gel composition of the present invention is generally within the range of about 40 to about 80% by weight of the invention and preferably about 40 to about 60% by weight of the composition. A humectant such as sorbitol, glycerin propylene glycol. PEG 600 is present in the vehicle of the present invention at a concentration of about 2 to about 15% by weight and preferably about 3 to about 8 by weight.

Examples of poly(ethylene oxides) useful in the practice of the present invention include PEG 2M, 5M, 7M, 14M, 23M, 45M and 90M commercially available from Union Carbide,

Danbury, Connecticut, ranging in molecular weight from 100,000 to 4 million. A

poly(ethylene oxide) preferred for use in the practice of the present invention is a poly(ethylene oxide) having a molecular weight of about 100,000. Such poly(ethylene oxide) or PEG 2M is a

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nonionic polymer of ethylene oxide having an average molecular weight of 100,000 and has the general formula:



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wherein n represents the number of repeating CH_2CH_2O groups.

Film forming agents useful in the practice of the present invention include Carbomers such as carboxymethylene polymers including acrylic acid polymers, and acrylic acid copolymers. Carboxypolymethylene is a slightly acidic vinyl polymer with active carboxyl groups. A
10 carboxypolymethylene preferred for use in the practice of the present invention is a water dispersible copolymer of acrylic acid cross-linked with approximately 0.75% to approximately 1.5% polyallyl sucrose that is sold under the trade designation Carbopol 934 or 974 by B.F. Goodrich. The Carbopol product is present in the liquid whitening composition of the present invention at a concentration of about 0.25 to about 1.5% by weight and preferably about 0.5 to
15 about 1.0% by weight.

Peroxide containing compounds which may be used as whitening agents in the practice of the present invention include hydrogen peroxide, urea peroxide and percarbonate salts such as sodium percarbonate persulfate salts such as sodium persulfate and perborate salts such as
20 sodium perborate. Most preferred is hydrogen peroxide. The peroxide containing compound is present in the liquid whitening gel compositions of the present invention at a concentration that will provide about 0.3 to about 12% by weight and preferably about 1 to about 10% by weight, active peroxide to the whitening gel formulation.

25 At the acid pH levels at which the composition of the present invention is maintained the Carbomer of the Carbopol type behaves like a liquid gel rather than a solid gel so that the Carbomer in combination with the poly(ethylene oxide) constituent provides thickness to the liquid product while maintaining a suitable consistency enabling the product to be painted on the tooth surface with a soft applicator brush.

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Polymeric opacifying particles useful in the practice of the present invention include olefin and fluoro polymers that may be incorporated in the liquid gel whitening compositions of the

- present invention which are unreactive with peroxide whitening agents present in the gel, and the gel once applied to the teeth forms a thin continuous film which can be visualized by the user. such polymers including polyethylene, polypropylene and ethylene/proylene copolymers, polytetrafluoro-ethylene and polyhexafluoropropene. The opacifying polymers are
- 5 incorporated in the liquid gel as powders having an average particle size in the range of about 0.5 to about 100 microns and preferably from about 1 micron to about 20 microns. Olefin polymers preferred in the practice of the present invention are polyethylene and polypropylene powders. Such powders are available from Micropowders, Inc. under the trade designation Micropoly 220 (polyethylene), Whitepoly 50, Microslip 519 (polytetrafluoroethylene).
- 10 The polymeric opacifying particles are incorporated in the liquid whitening gel compositions of the present invention at concentrations of about 1 to about 30% by weight and preferably about 3 to about 9% by weight without affecting the stability of the peroxide whitening agent present in the liquid whitening gel.
- 15 Titanium dioxide and titanium dioxide coated mica particles having an average particle size range of about 0.5 to about 75 microns and preferably about 5 to about 60 microns are also useful opacifying agents in liquid whitening gel of the present invention except that the concentration of these particles in the gel composition are used in the range of up to 1.0%, and preferably between 0.15% to 1.0% by weight. Higher concentration levels of the titanium
- 20 dioxide and titanium dioxide coated mica particles have tendency to destabilize the peroxide whitening agent during storage of the liquid whitening gel.

- The liquid whitening gel composition of the present invention may also contain a flavoring agent. Flavoring agents that are used in the practice of the present invention include natural or
- 25 synthetic essential oils as well as various flavoring aldehydes, esters, alcohols, and similar materials. Examples of the essential oils include oils of spearmint, peppermint, wintergreen, sassafras, clove, sage, eucalyptus, marjoram, cinnamon, lemon, lime, grapefruit, and orange. Also useful are such chemicals as menthol, carvone, and anethole. Of these, the most commonly employed are the oils of peppermint, spearmint and wintergreen. The flavoring
- 30 agent is incorporated in the whitening liquid composition of the present invention at a concentration of about 0.1 to about 2% by weight and preferably about 0.1 to about 0.5% by weight.

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The liquid gel whitening composition of the present invention is initially prepared in the form of a liquid varnish and applied as such to the users teeth as by painting the teeth with a soft applicator. After application by the user, the alcohol and water vehicle constituents rapidly evaporate to leave a film or coating of a thick liquid gel on the teeth to which the varnish has been applied. The deposited film is comprised of the poly(ethylene oxide) constituent, the Carbomer constituent, the opacifier and the peroxide whitening agent or agents. The presence of the poly(ethylene oxide) and Carbomer constituent combination permits a slow release of the peroxide agent or agents to the applied tooth site, providing prolonged whitening treatment of the site.

The deposited film of liquid gel contains no ingredients imparting thereto an unacceptable taste or texture, rendering it unpleasant to the user and adheres strongly to tooth enamel. After application to the teeth, the composition dries in a relatively short time period, for example, 20 to 60 seconds to yield a strongly adherent, even layered opaque film which can be easily visualized by the user and distinguished from the tooth surface during application and while in place. The film gel is sufficiently strong and adherent enough to remain on the teeth for a period of time, for example up to 30 minutes to effect a whitening result and will resist the forces commonly applied by the lips and tongue. While the film is in place, the user is to refrain from mastication. The film can be removed as and when required, at will, by an employment of standard oral hygiene procedures such as brushing or by rinsing with an alcoholic mouthwash. While in place the film releases agents contained therein at a slow, relatively constant rate and in concentration sufficient effectively to effect stain removal from the teeth.

The whitening benefit imparted by the whitening gel of the present invention may be enhanced by brushing the teeth, before or after the application of the whitening gel, with a whitening toothpaste containing peroxide whitening agents or stain removing silica or alumina abrasives or a rinse composition having an alkaline pH as disclosed in US 6,174,516.

The liquid whitening gel compositions of the present invention are prepared by adding and mixing the ingredients of the composition in a suitable vessel such as a stainless steel tank

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provided with a mixer. In the preparation of the liquid whitening composition, the ingredients are advantageously added to the mixer in the following order: solvent film forming agent, humectant and peroxide compound and any desired flavoring. The ingredients are then mixed to form a homogeneous dispersion/solution. Thereafter an opacifier such as polyethylene, 5 polytetrafluoroethylene or titanium dioxide coated mica particles are added to the solution. The present invention is illustrated by the following example but is not to be limited thereby.

Example

10 A liquid whitening gel base formula having a pH of 4.0 was prepared having the ingredients listed in Table I below:

TABLE I	
Liquid Whitening Gel Base Formula	
Ingredient	% by Weight
Water	10.07
Carbopol 974	1.00
95% Ethyl alcohol	34.8
Glycerin	5.00
PEG 600	10.00
PEG 2M	14.00
Hydrogen peroxide	25.00
85% Phosphoric acid	0.05
Monobasic sodium phosphate	0.05
Total	100.00

A series of liquid whitening gels was prepared in which the base formula of Table I was modified by either (1) 2 to 5% by weight a polyethylene (PE) powder having an average 15 particle size of 6 to 8 microns: (2) 1 to 5% by weight of a polytetrafluoroethylene (PTFE) powder having particle size of 5 to 6 microns: (3) 0.8 to 2.5% by weight polypropylene powder having a particle size of 4 to 50 microns: (4) 2 to 5% by weight of the PE powder and 0.11 to 0.4% by weight of titanium dioxide powder having a particle size of 10 to 45 microns. The amounts of the polymer and inorganic titanium powders incorporated in the base formula are 20 recorded in Tables II and III.

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TABLE II							
Liquid Whitening Gel Formula	A (Wt.%)	B (Wt.%)	C (Wt.%)	D (Wt.%)	E (Wt.%)	F (Wt.%)	G (Wt.%)
Base Formula	99.0	98.0	95.0	98.0	95.0	99.2	97.5
+ PE	--	--	--	2.0	5.0	--	--
+ PTFE	1.00	2.0	5.0	--	--	--	--
+ PP	--	--	--	--	--	0.8	2.5

TABLE III						
Liquid Whitening Gel Formula	GA (Wt.%)	II (Wt.%)	I (Wt.%)	J (Wt.%)	K (Wt.%)	L (Wt.%)
Base Formula	99.5	97.90	97.80	97.60	94.90	99.5
+ PE	--	2.00	2.00	2.00	5.00	--
+ TiO ₂	--	0.10	0.20	0.40	0.10	--
+ TiO ₂ coated mica	0.5	--	--	--	--	--

The shelf stability of the liquid whitening gel formulations A-K, packaged in plastic bottles was determined by the percent of active oxygen present in the whitening liquid after a two week exposure of plastic bottle packaged whitening compositions to a temperature of 120°F (49°C).

- 5 Active oxygen analysis was performed using a standard iodometric titration method. The stability results are recorded in Table IV below.

TABLE IV				
Active Oxygen Analysis				
Liquid Whitening Gel Formula	Initial	Final	% Loss*	
A	9.07	8.95	None	
B	9.22	8.78	None	
C	8.79	8.28	5.9%	
D	8.83	9.08	None	
E	8.76	8.57	None	
F	9.01	8.88	None	
G	8.50	8.97	None	
GA	9.05	8.95	None	
H	8.97	9.13	None	
I	9.10	8.85	None	
J	8.63	8.70	None	
K	8.61	8.41	None	
L	9.05	8.95	None	

- 10 *Loss of <5% is considered experimental error.

The active oxygen analysis results recorded in Table IV indicate that the liquid whitening gel of the present invention retained sufficient peroxide content to be an efficacious whitening composition even after two (2) weeks of storage at the elevated temperature of 120°F (49°C).

- 15 Past experience with this temperature stability test indicated that when peroxide loss is less than 25% after two weeks at 120°F, a minimum shelf life of six (6) months can be expected.

Example II

Following in the procedure of Example I, a liquid whitening gel designated "Gel L" was prepared having the ingredients listed in Table V in which 0.38% by weight of titanium dioxide particles having a particle size of 10 to 45 microns was incorporated. Gel L packaged in plastic bottles after two (2) weeks at 120°F active oxygen analysis indicated an oxygen loss of less than 5%.

TABLE V	
Liquid Gel Formula	Gel L (W%)
Carbomer	1.00
PEG 600	9.96
BHT	0.03
Glycerin	4.98
Water	17.00
Ethyl alcohol	34.67
PEG 2M	13.95
H ₂ O ₂ (35%)	17.93
Sodium Phosphate	0.05
Phosphoric acid	0.05
Titanium dioxide	0.38

For purposes of comparison, the procedure of the Example II was repeated except the base formula was modified with 5% by weight TiO₂ designated "Gel M". After two (2) weeks at 120°F, active oxygen analysis indicated that Gel M had an unacceptable oxygen loss of 41.24%.

For purposes of further comparison, a liquid whitening gel formula of the prior art (US 5,425,953) designated Gel N, the ingredients of which are listed in Table VI, below. Gel N was modified with 0.19 and 0.38% by weight of titanium oxide particles. These gels designated Liquid Gel Formula O and P were also prepared using the ingredients listed in Table VI below.

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TABLE VI		
Liquid Whitening Gel Formulation	N (Wt. %)	O (Wt. %)
Water	28.95	29.27
Ethanol	50.50	51.80
Hydroxypropyl cellulose	12.45	12.45
Carbamide peroxide	8.00	6.00
Calcium EDTA	0.10	0.10
Titanium dioxide	0.00	0.38

The shelf stability of whitening gels N and O packaged in plastic bottles was determined as the percent peroxide recovered from the gels after a 2 week exposure at 120°F (49°C). Percent peroxide recovery was performed using standard iodometric titration method. The stability

 5 results are recorded in Table VII below.

TABLE VII			
LIQUID WHITENING GEL STABILITY			
Liquid Whitening Gel Formula	Initial Wt. % H ₂ O ₂	Wt. % H ₂ O ₂ After 2 Weeks at 120°F	% Recovery of H ₂ O ₂
N	7.89	6.00	76
O	5.54	0.67	12

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Claims

5 What is claimed is:

1. A stable opaque liquid tooth whitening gel composition suitable for application to teeth in the oral cavity comprising a peroxide containing compound dispersed in an orally acceptable vehicle containing a solvent, a film forming agent and an opacifier selected from organic and inorganic powders having a particle size of about 0.5 to 100 microns.
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2. The composition of claim 1 wherein the vehicle is comprised of water and a monohydric alcohol.
- 15 3. The composition of claim 1 wherein the film forming agent is a mixture of a poly(ethylene) oxide and a Carbomer.
4. The composition of claim 3 wherein the poly(ethylene oxide) is present in the composition in an amount from about 5 to about 50 weight %.
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5. The composition of claim 3 wherein the Carbomer is a cross-linked acrylic acid copolymer.
6. The composition of claim 3 wherein the Carbomer is present in an amount from about 0.25 to about 1.5 weight %.
25
7. The composition of claim 1 wherein the peroxide containing compound is urea peroxide or hydrogen peroxide.
- 30 8. The composition of claim 1 wherein the peroxide containing compound is present in the composition at a concentration that will provide about 0.3 to about 12% by weight peroxide to the composition.
9. The composition of claim 1 wherein the concentration of the peroxide containing compound is present in the composition at a concentration that will provide about 1 to about 10% by weight peroxide to the composition.
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- 63 - 67
10. The composition of claim 1 wherein the opacifier is an organic polymer having an average particle size in the range of 1 to about 100 microns.
- 5 11. The composition of claim 1 wherein the organic opacifier has a particle size of about 1 to about 20 microns.
12. The composition of claim 10 wherein the organic polymer is polyethylene.
- 10 13. The composition of claim 10 wherein the organic polymer is polypropylene.
14. The composition of claim 10 where the organic polymer is polytetrafluoroethylene.
- 15 15. The composition of claim 1 wherein the organic opacifier is present in the composition at a concentration of about 1 to about 10% by weight.
16. The composition of claim 1 wherein the inorganic opacifier has a particle size of about 5 to about 60 microns.
- 20 17. The composition of claim 1 wherein the inorganic opacifier is titanium dioxide or titanium dioxide coated mica.
18. The composition of claim 1 wherein the inorganic opacifier is present in the composition at a concentration of about 0.10% to about 1% by weight.
- 25 19. The method for whitening teeth which comprises:
- (a) preparing a liquid gel tooth whitening composition of claim 1;
- (b) painting the composition into contact with the teeth to be whitened;
- 30 (c) maintaining the composition in contact with the teeth to effect tooth whitening.

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ABSTRACT

5

A storage stable liquid peroxide whitening gel composition opacified with an organic and inorganic particles.

GENERAL POWER OF ATTORNEY

(for several international applications filed under the Patent Cooperation Treaty)

(PCT Rule 90.5)

The undersigned person(s):

(Family name followed by given name; for a full legal entity, full official designation. The address must include postal code and name of country.)

COLGATE-PALMOLIVE COMPANY

300 Park Avenue

New York, New York 10022

United States of America

hereby appoint(s) the following person as:

☒ agent☐ common representative

Name and address

(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

Martin B. Berancik, Henry S. Goldfine, Bernard Lieberman, Michael J. McGrath, Rosemary M. Milano, Richard E. Nanfeldt, Ellen Park and Clifford Wilkins of

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United States of America

to represent the undersigned before

☒ all the competent International Authorities☐ the International Searching Authority only☐ the International Preliminary Examining Authority only

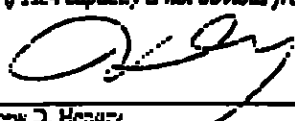
in connection with any and all international applications filed by the undersigned with the following Office

RO/US

as receiving Office

and to make or receive payments on behalf of the undersigned.

Signature(s) (where there are several persons, each of them must sign; next to each signature, indicate the name of the person signing and the capacity in which the person signs. If such capacity is not obvious from reading this power:



 Andrew D. Henary
 Senior Vice President, General Counsel and
 Secretary
Date: October 15, 2003

PCT (ANNEX - FEE CALCULATION SHEET)

Original (for SUBMISSION)

(This sheet is not part of and does not count as a sheet of the international application)

0	For receiving Office use only		
0-1	International Application No.		
0-2	Date stamp of the receiving Office		
0-4	Form PCT/RO/101 (Annex) PCT Fee Calculation Sheet		
0-4-1	Prepared Using	PCT-SAFE [EASY mode] Version 3.50 (Build 0002.153)	
0-8	Applicant's or agent's file reference	7091-00	
2	Applicant	COLGATE-PALMOLIVE COMPANY	
12	Calculation of prescribed fees	fee amount/multiplier	Total amounts (USD)
12-1	Transmittal fee T	⇒	300
12-2-1	Search fee S	⇒	1818
12-2-2	International search to be carried out by EP		
12-3	International filing fee (first 30 sheets) I1	1035	
12-4	Remaining sheets	0	
12-5	Additional amount (X) 0		
12-6	Total additional amount I2	0	
12-7	I1 + I2 = I	1035	
12-12	EASY Filing reduction R	-74	
12-13	Total international filing fee (I-R)	⇒	961
12-14	Fee for priority document		
	Number of priority documents requested	1	
12-15	Fee per document (X) 20		
12-18	Total priority document fee: P	⇒	20
12-17	TOTAL FEES PAYABLE (T+S+I+P)	⇒	3099
12-19	Mode of payment	authorization to charge deposit account	
12-20	Deposit account instructions		
	The receiving Office	United States Patent and Trademark Office (USPTO) (RO/US)	
12-20-1	Authorization to charge the total fees indicated above.	✓	
12-20-2	Authorization to charge any deficiency or credit any overpayment in the total fees indicated above.	✓	
12-20-3	Authorization to charge the fee for priority document.	✓	
12-21	Deposit account No.	03-2457	
12-22	Date	15 January 2004 (15.01.2004)	
12-23	Name and signature	DUNNE, Elizabeth, M. <i>Elizabeth M. Dunne</i>	

INTERNATIONAL SEARCH REPORT

International Application No.
PCT/02004/000979

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A61K7/20 A61K7/16

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 7 A61K

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 practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 934 000 A (J. BARTH) 20 January 1976 (1976-01-20) column 4, lines 66-68; claims; examples 7.8	1,16-19
Y		1-9, 16-19
X	US 3 944 661 A (D. COLODNEY ET AL) 16 March 1976 (1976-03-16) column 3, lines 44-46; examples 2,3 column 4, line 65 - column 5, line 11	1,16-19
X	US 3 954 961 A (CORDON MARTIN ET AL) 4 May 1976 (1976-05-04) column 1, lines 42-62; claims: examples 1.2.4 column 4, lines 25-27	1,16-19
	-/-	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in 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 document but published on or after the international filing date
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- *O* document referring to an oral disclosure, use, exhibition or other matters
- *P* document published prior to the international filing date but later than the priority date claimed

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Date of the actual completion of the international search

19 July 2004

Date of mailing of the international search report

03/08/2004

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/US2004/000979

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 957 968 A (M. CORDON) 18 May 1976 (1976-05-18) column 4, lines 18-20: example 5	1,16-19
X	US 4 075 316 A (M. CORDON) 21 February 1978 (1978-02-21) column 4, lines 14-16: example 5	1,16-19
Y	US 6 331 291 B1 (W. GLACE ET AL.) 18 December 2001 (2001-12-18) column 13, lines 24-34; examples	1-9, 16-19
A	US 6 447 757 B1 (J. ORLOWSKI ET AL.) 10 September 2002 (2002-09-10) column 4, lines 57-59: claims; examples	1-19
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Information on patent family members

International Application No

PCT/002004/000979

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No.

PCT/JP2004/000979

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PATENT COOPERATION TREATY

PCT

NOTIFICATION CONCERNING
SUBMISSION OR TRANSMITTAL
OF PRIORITY DOCUMENT

(PCT Administrative Instructions, Section 411)

From the INTERNATIONAL BUREAU

To:

PARK, Ellen, K.
Colgate-Palmolive Company
909 River Road
P.O. Box 1343
Piscataway, NJ 08855
United States of America

Date of mailing (day/month/year) 21 April 2004 (21.04.2004)	
Applicant's or agent's file reference 7091-00	IMPORTANT NOTIFICATION
International application No. PCT/US2004/000979	International filing date (day/month/year) 15 January 2004 (15.01.2004)
International publication date (day/month/year) Not yet published	Priority date (day/month/year) 15 January 2003 (15.01.2003)
Applicant COLGATE-PALMOLIVE COMPANY	

- By means of this Form, which replaces any previously issued notification concerning submission or transmittal of priority documents, the applicant is hereby notified of the date of receipt by the International Bureau of the priority document(s) relating to all earlier application(s) whose priority is claimed. Unless otherwise indicated by the letters "NR", in the right-hand column or by an asterisk appearing next to a date of receipt, the priority document concerned was submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b).
- (If applicable) The letters "NR" appearing in the right-hand column denote a priority document which, on the date of mailing of this Form, had not yet been received by the International Bureau under Rule 17.1(a) or (b). Where, under Rule 17.1(a), the priority document must be submitted by the applicant to the receiving Office or the International Bureau, but the applicant fails to submit the priority document within the applicable time limit under that Rule, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.
- (If applicable) An asterisk (*) appearing next to a date of receipt, in the right-hand column, denotes a priority document submitted or transmitted to the International Bureau but not in compliance with Rule 17.1(a) or (b) (the priority document was received after the time limit prescribed in Rule 17.1(a) or the request to prepare and transmit the priority document was submitted to the receiving Office after the applicable time limit under Rule 17.1(b)). Even though the priority document was not furnished in compliance with Rule 17.1(a) or (b), the International Bureau will nevertheless transmit a copy of the document to the designated Offices, for their consideration. In case such a copy is not accepted by the designated Office as priority document, Rule 17.1(c) provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.

Priority date	Priority application No.	Country or regional Office or PCT receiving Office	Date of receipt of priority document
15 Janu 2003 (15.01.2003)	10/342,076	US	19 April 2004 (19.04.2004)

RECEIVED

PATENT DEPARTMENT

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland

Facsimile No. (41-22) 338.89.70

Elisabeth KÖNIG (Fax 338 8970)

Telephone No. (41-22) 338 8748

PATENT COOPERATION TREATY

From the RECEIVING OFFICE

PCT

NOTIFICATION OF THE INTERNATIONAL
APPLICATION NUMBER AND OF THE
INTERNATIONAL FILING DATE

(PCT Rule 20.5(c))

To:

ELLEN K. PARK
COLGATE-PALMOLIVE COMPANY
909 RIVER ROAD
P.O. BOX 1343
PISCATAWAY, NEW JERSEY 08855Date of mailing
(day/month/year)

22 MARCH 2004

Applicant's or agent's file reference

7091-00

IMPORTANT NOTIFICATION

International application No.

PCT/US2004/000979

International filing date (day/month/year)

15 Jan 2004

Priority date (day/month/year)

15 Jan 2003

Applicant

COLGATE-PALMOLIVE COMPANY

Title of the invention

LIQUID TOOTH WHITENING GEL

1. The applicant is hereby notified that the international application has been accorded the international application number and the international filing date indicated above.

2. The applicant is further notified that the record copy of the international application:



was transmitted to the International Bureau on 22 MARCH 2004



has not yet been transmitted to the International Bureau for the reason indicated below and a copy of this notification has been sent to the International Bureau*:



because the necessary national security clearance has not yet been obtained.



because (reason to be specified):

* The International Bureau monitors the transmittal of the record copy by the receiving Office and will notify the applicant (with Form PCT/IB/301) of its receipt. Should the record copy not have been received by the expiration of 14 months from the priority date, the International Bureau will notify the applicant (Rule 22.1(c)).

Name and mailing address of the receiving Office

Mail Stop PCT, Commissioner for Patents
P.O. Box 1450, Alexandria, VA 22313-1450

Facsimile No. 703-305-3230

Authorized officer

Valerie Jackson

Telephone No. 703-305-3756

Form PCT/RO/105 (July 1992; reprint January 2004)

23 23



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Page 1

I, NORMAN GOODMAN, County Clerk and Clerk of the Supreme Court of the State of New York, in and for the County of New York, a Court of Record, having by law a seal, DO HEREBY CERTIFY pursuant to the Executive Law of the State of New York, that

Rosie S. Dizon

whose name is subscribed to the annexed affidavit, deposition, certificate of acknowledgment or proof, was at the time of taking the same a NOTARY PUBLIC in and for the State of New York duly commissioned, sworn and qualified to act as such; that pursuant to law, a commission or a certificate of his official character, with his autograph signature has been filed in my office; that at the time of taking such proof, acknowledgment or oath, he was duly authorized to take the same; that I am well acquainted with the handwriting of such NOTARY PUBLIC or have compared the signature on the annexed instrument with his autograph signature deposited in my office, and I believe that such signature is genuine.

IN WITNESS WHEREOF, I have hereunto set my hand affixed my official seal this

OCT 30 2001

FEE PAID \$3.00


County Clerk and Clerk of the Supreme Court, New York County



Apostille

(Convention de La Haye du 5 Octobre 1961)

1. Country: **United States of America**

This public document

2. has been signed by **Norman Goodman**

3. acting in the capacity of **County Clerk**

4. bears the seal/stamp of the county of **New York**

Certified

5. At New York, New York

6. the 30th day of October 2001

7. by Special Deputy Secretary of State, State of New York

8. No. NYC-9786540B

9. Seal/Stamp

10. Signature

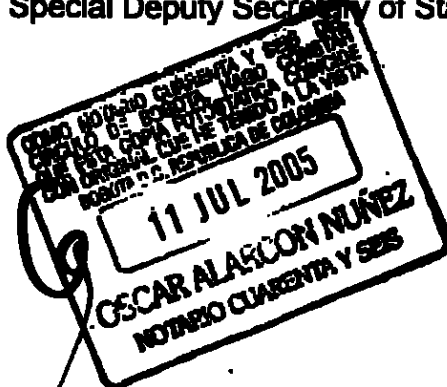


04135501.RSL (REV: 2/9/00)

Aqil M. Qureshi

Aqil M. Qureshi

Special Deputy Secretary of State



Brigard & Castro

SANTA FE DE BOGOTA - COLOMBIA

PODER

POWER OF ATTORNEY

Yo (nosotros)

domiciliado(a) en

Nombre(amos) a: RAMIRO CASTRO DUQUE y/o JUAN PABLO CADENA SARMIENTO y/o BEATRIZ JARAMILLO S.

de Santafé de Bogotá, Colombia, apoderados verdaderos y legales con poder especial, amplio y suficiente, para recabar de las oficinas y autoridades colombianas administrativas, judiciales y de policía, en cualquier instancia o recurso, la obtención de patentes de invención, registros de marcas, diseños industriales, modelos de utilidad, depósitos de nombres comerciales y enseñas, registros de derechos de autor y contratos sobre los mismos, obtención de certificados de obtentor de variedades vegetales, así como sus prórrogas, renovaciones, anotaciones de traspaso, cambios de nombre y/o domicilio; elaborar, tramitar y registrar contratos de licencia y licencias de cualesquiera clases; intervenir como demandante o como demandado en cualquier instancia y recurso ante cualquier juez, corporación, funcionario público o autoridad en acciones judiciales, administrativas y de policía tales como: oposiciones al registro de marcas; acciones de caducidad, cancelación y nulidad; acciones reivindicatorias, acciones derivadas de la obtención o explotación de licencias; acciones de competencia desleal; demandas penales; acciones de indemnización de perjuicios, reclamos y observaciones a solicitudes de patentes, diseños industriales, modelos de utilidad, en el ejercicio de medidas cautelares y en otras acciones o medidas similares.

A este efecto, lo(a) facuto(amos) para elevar solicitudes, formular descripciones, enmiendas, protestas, declaraciones, oposiciones, cancelaciones, nulidades, apelaciones y reclamos; pagar impuestos, cuotas y gravámenes prescritos por la ley; renunciar o desistír a derechos concedidos o en curso de concesión; recibir toda clase de documentos y valores, dando el descargo respectivo, y, en general, para dar ante dichas autoridades todos los pasos necesarios al efecto indicado y tomar todas las medidas que creyeren conducentes al resguardo de mis (nuestros intereses), con todas las demás facultades legales necesarias.

Este poder comprende la facultad de ratificar, confirmar en mí, (nuestro) nombre lo anterior, como la facultad de desistír, transigir, comprometer y substituir en todo o en parte, a favor de nosotros, a RAMIRO CASTRO DUQUE y/o JUAN PABLO CADENA SARMIENTO y/o BEATRIZ JARAMILLO S. domiciliado(a) en la Carrera 7 No. 16-58, Piso 9, mis (nuestros) apoderados en Colombia, con facultades para recibir notificaciones y designar abogados judiciales o extrajudiciales.

I (We)

COLGATE-PALMOLIVE COMPANY, a Delaware corporation, of 300 Park Avenue, New York, New York 10022, U. S. A.

Hereby appoint RAMIRO CASTRO DUQUE and/or JUAN PABLO CADENA SARMIENTO and/or BEATRIZ JARAMILLO S.

of Santafé de Bogotá, Colombia, my (our) true and lawful attorneys with special, ample and sufficient power to obtain from the Colombian administrative, judicial and police offices and authorities, in any instance, privileges of patents of invention, registration of trademarks, industrial designs and utility models; registration of commercial names and emblems, registration of copyright and contracts related thereto, certificates of obtainers of vegetable varieties, as well as extensions, renewals and records of assignments and changes of name and domicile related to the above; to prepare, prosecute and register licenses of any kind; to act as plaintiff or defendant in any instance or recourse, and before any judge, corporation, public official or authority in judicial, administrative and police actions, such as: oppositions to the registration of trademarks; caducity, cancellation and nullity actions; claim actions derived from the obtaining or exploitation of licenses, action of unfair competition; penal demands, actions of indemnities for damages; claims or observations on patent applications, models, industrial designs and utility models; in the exercise of preventive measures; and in other actions or similar proceedings.

To that effect they are hereby empowered to file applications, formulate descriptions, amendments, protests, declarations, oppositions, cancellations, nullities, appeals and claims; to pay taxes, quotas and assessments prescribed by law, renounce or waive the rights granted or in the process of granting; to receive all kind of documents and valuables, giving the respective release of receipt, and, in general, to take before said authorities the necessary steps to such effect, and any measures that they may deem pertinent to the safeguard of my (our) interests) with all the other necessary legal powers.

This power includes the faculty to ratify or confirm, confirm in me, (our) name the above, and to substitute this power in whole or in part and to make substitutions. At the same time I (We) hereby appoint RAMIRO CASTRO DUQUE and/or JUAN PABLO CADENA SARMIENTO and/or BEATRIZ JARAMILLO S. domiciled in Carrera 7 No. 16-58, Piso 9, my (our) attorneys in fact, with powers to receive notifications and to designate judicial and extrajudicial attorneys.

Otorgado y firmado en

hoy de

Firma

Nombre

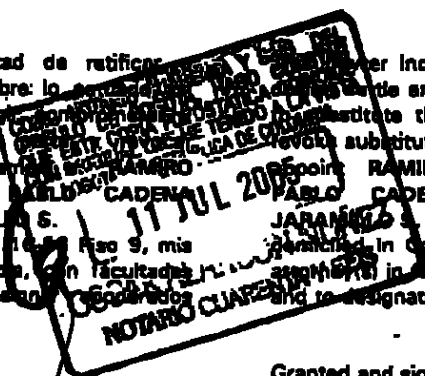
Granted and signed in New York, N.Y.

this OCT 29 2001 day of 2001

Signature

Name

VICE PRESIDENT &
ASSOCIATE GENERAL COUNSEL
TRADEMARK



76
70

CERTIFICADO NOTARIAL (SOCIEDAD)

NOTARIAL CERTIFICATE (CORPORATION)

Hoy de de
personalmente compareció ante mí

a quien conozco, y de quien me consta que firmó el
anterior documento, debidamente juramentado declaró
y dijo que él es

de

Sociedad legalmente constituida para un periodo de
duración que aún no ha terminado; que el declarante
está autorizado por dicha compañía para conferir este
poder; que el sello estampado por él al pie de tal poder,
en nombre y representación de la expresada compañía,
es el sello oficial de ella; y que el objeto para el cual se
confiere este instrumento está comprendido dentro del
de la sociedad.

El suscrito Notario hace constar que tuvo a la vista las
pruebas de la existencia de la sociedad.

Estado de
con fecha

y que quien confiere el presente poder es su
representante.

On this OCT 29 2001 day of of
personally appeared before me

to me known, and known to me to be the person who
signed the foregoing instrument who being by me duly
sworn did depose and say that he (she) is the

VICE PRESIDENT &
ASSOCIATE GENERAL COUNSEL
TRADEMARK
COLGATE-PALMOLIVE COMPANY

Which is legally constituted for a term not yet expired,
that deponent is authorized by said Company to confer
this Power of Attorney: that the seal affixed hereunto
by the deponent in the name and on behalf of said
Corporation is the corporate seal thereof; and that the
purpose for which this instrument is granted is
included within the corporate purpose of company.

The undersigned Notary Public attests that he has seen
the documents that prove the existence of the
company.

State of Delaware
under date of May 13, 1997

and that he (she) who grants the present power of
attorney is its Representative.

Notario Público (sello)

Rosie S. Dizon
Notary Public

CERTIFICADO NOTARIAL
PERSONA NATURAL

ROSIE S. DIZON
Notary Public, State of New York
No. 31-4941117
Qualified in New York County
Commission Expires Aug. 15, 2002

COUNTY

Hoy de de , compareció(eron)
ante mí

On this OCT 29 2001 day of of
personally appeared before me

SCOTT E THOMPSON

a quien (es) conozco y me consta que es(son) la(s)
persona(s) firmante(s) del documento que precede y
quien(es) declara(n) que otorgan el mismo para los
fines y propósitos que en él se expresan.

who is/are known to me and who I attest, executed
the foregoing document and he (they) declared he
she (they) is/are the person(s) who executed the document for the
purpose(s) expressed.

Notario Público (sello)

11 JUL 2005
OSCAR ALARCON NUNEZ
NOTARIO CUARENTA Y SEIS

Rosie S. Dizon
Notary Public (sello)

LEGALIZACION POR APOSTILLA ES REQUERIDA.

LEGALIZATION BY Notary Public, State of New York
No. 31-4941117
Qualified in New York County
Commission Expires Aug. 15, 2002

xx ~~st~~

1. País: ESTADOS UNIDOS DE AMERICA
Este documento público

3. actuando en capacidad de Oficial del Condado

CERTIFICADO

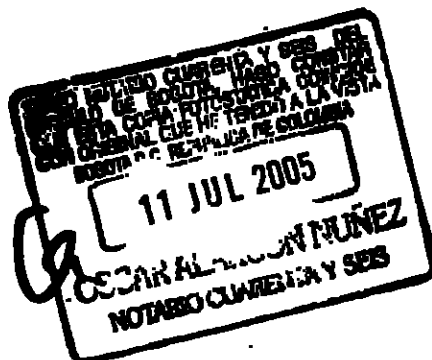
6. el 30 de Octubre de 2001

8. No. NYC-9786540B

10. Firma

Aquil M. Qureshi

Secretario de Estado Encargado Especial



20

Estado de New York)
) ss.
Condado de New York)

Yo, NORMAN GOODMAN, Oficial del Condado y Oficial de la Corte Suprema del Estado de New York, en y para el Condado de New York, un Tribunal de Registros, que tiene un sello por ley.

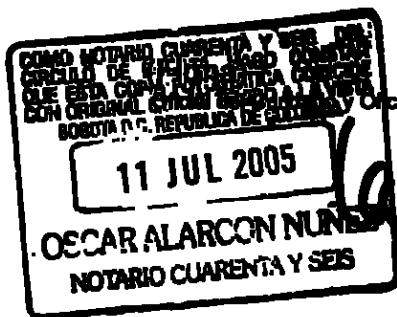
ROSIE S. DIZON

cuyo nombre está suscrito al acta notarial, deposición, certificado de reconocimiento o prueba, era al momento de tomar el mismo NOTARIO PÚBLICO en y para el Estado de New York debidamente comisionado, juramentado y calificado para actuar como tal; que de acuerdo con la ley, una comisión o un certificado de este carácter oficial, con su firma autógrafa ha sido presentada en mi oficina; que al momento de tomar tal prueba, reconocimiento o juramento, estaba debidamente autorizado para tomar el mismo; que estoy bien familiarizado con la escritura de tal NOTARIO PÚBLICO o he comparado la firma en el instrumento anexo con la firma autógrafa depositada en mi oficina, y creo que tal firma es genuina.

COMO TESTIGO DE LO MISMO, he fijado al presente mi firma t mi sello oficial este

30 de Octubre de 2001

TARIFA PAGADA \$3.00



Oficial de la Corte Suprema, Condado de New York



CAVIRI

ORGANIZACION ELECTORAL
REGISTRADURIA NACIONAL DEL ESTADO CIVIL

REGISTRO CIVIL DE DEFUNCION

Indicativo
Serial

5562241

Datos de la oficina de registro							
Clase de oficina: ..	Registraduría	Notaría	☒	Consulado	Corregimiento	Ins. de Policía	Código 9795
País - Departamento - Municipio - Corregimiento o Inspección de Policía							
COLOMBIA CUNDINAMARCA BOGOTA D.C.				NOTARIA 32.			

Datos del inscrito	
Apellido y nombres completos	
CASTRO DUQUE RAMIRO	
Documento de identificación (Clase y número)	Sexo (en Letras)
C.C.No. 170322 DE BOGOTA	MASCULINO

Datos de la defunción			
Lugar de la Defunción País - Departamento - Municipio - Corregimiento o Inspección de Policía			
COLOMBIA CUNDINAMARCA BOGOTA D.C.			
Fecha de la defunción		Hora	Número de certificado de defunción
Año 2004	Mes 10	Día 30 04:10 PM	A- 2094375.
Lugar donde se profirió la sentencia		Fecha de la sentencia	
-----		-----	
Documento presentado		Nombre y cargo del funcionario	
Autorización Judicial ☐	Certificado Médico ☒	ANDRES DIAZ CAMPOS.- R. M. No. 79982662	

Datos del denunciante	
Apellido y nombres completos	
EDGAR DAVID REGINO II.	
Documento de identificación (Clase y número)	Firma
C.C.No. 15.030.154 DE LORICA/ CORDOBA	

Primer testigo	
Apellido y nombres completos	

Documento de identificación (Clase y número)	Firma
-----	-----

Segundo testigo	
Apellido y nombres completos	

Documento de identificación (Clase y número)	Firma
-----	-----

Fecha de inscripción		Notario y Firma del Registrador que autoriza	
Año 2004	Mes 11	Día 02	BLANCA SOTO GONZALEZ RESTREPO

PADRES: ALFON SO CASTRO		ESPACIO PARA NOTAS	
MERCEDES DUQUE.		-----	

ORIGINAL PARA LA OFICINA DE REGISTRO

NOTARIA 32

DEL CÍRCULO DE SANTA FE DE BOGOTÁ, D.C.

Germán Eleázar Moreno L.
Notario

COMO SECRETARIO DELEGADO DE LA NOTARIA TREINTA Y DOS (32) DEL
CÍRCULO DE BOGOTÁ

CERTIFICO QUE:

La presente acta es fiel copia del original tomada del registro
civil de defunciones serial indicativo número: **ST 6224**
que expido en la ciudad de Bogotá D.C. hoy: **05 NOV. 2004**

SECRETARIA DELEGADA



REQUISITOS NECESARIOS PARA PRESENTACION Y AGILIZACION DEL TRAMITE

	USUARIO	RADICADOR
PATENTE DE INVENCION ✓	()	()
MODELO DE UTILIDAD	()	()
- Indicación de que se solicita la concesión de una patente.	()	()
- Datos de identificación del solicitante o de la persona que se presenta la solicitud.	()	()
- Descripción de la invención.	()	()
- Dibujos de ser estos pertinentes.	()	()
- Comprobante de Pago de las tasas establecidas.	()	()
COMPLETA ✓	INCOMPLETA ()	()
 DISEÑO INDUSTRIAL ()		
- Indicación de que se solicita el registro de Diseño Industrial	()	()
- Datos de identificación del solicitante o de la persona que presenta la solicitud.	()	()
- Representación gráfica o fotográfica del Diseño Industrial o muestra del Material que incorpora el diseño.	()	()
- Comprobante de pago de las tasas establecidas	()	()
COMPLETA ()	INCOMPLETA ()	()
 ESQUEMA DE TRAZADO ()		
- Indicación de que solicita el registro de un Esquema de trazado.	()	()
- Datos de identificación del solicitante o de la persona que presenta La solicitud.	()	()
- Representación gráfica del esquema de trazado	()	()
- Comprobante de pago de las tasas establecidas.	()	()
COMPLETA ()	INCOMPLETA ()	()

Firma Responsable:

Fecha:

81
81

REPUBLICA DE COLOMBIA
SUPERINTENDENCIA DE INDUSTRIA Y C MERCI

Bogotá,
2020

Doctor(a)
CADENA SARMIENTO JUAN PABLO
Apoderado(a) y/o Representante de
COLGATE PALMOLIVE COMPANY

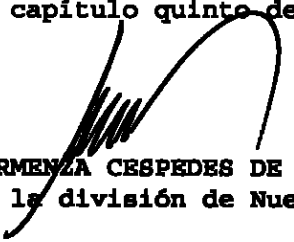
REFERENCIA	Radicación No.	05 78830
	Solicitud internacional	US2004/000979
	Fecha inicio fase nacional	15/08/2005
	Trámite	11
	Evento	379
	Actuación	430
	Oficio No.	8726

Como resultado del examen de forma, realizado con fundamento en el artículo 38 de la Decisión 486 de la Comisión de la Comunidad Andina, artículo 27 del Tratado de Cooperación en Materia de Patentes (PCT), regla 51 bis del Reglamento y Circular Única de la Superintendencia de Industria y Comercio, se le comunica que:

1. Si fuere el caso, el solicitante debe adjuntar la traducción de los anexos del reporte de examen preliminar internacional (Art. 36.2.b) del tratado PCT.

En cumplimiento de lo dispuesto por el artículo 39 de la Decisión 486, se le requiere para que dentro del término de dos meses siguientes a la fecha de notificación del presente oficio, complete los requisitos anteriormente enunciados. En caso de no dar cumplimiento al presente requerimiento, la solicitud se considerará abandonada y perderá su prelación.

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


ALIX CARMENZA CESPEDES DE VERGEL
Jefe de la división de Nuevas Creaciones

El anterior requerimiento fue notificado por fijación en lista de
fecha 23 SET. 2005
adpal