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(71) Applicant: THE PROCTER & GAMBLE COMPANY  
[US/US]; One Procter & Gamble Plaza, Cincinnati, Ohio  
45202 (US).(72) Inventors: GLENN, Robert, Wayne, Jr.; One Procter &  
Gamble Plaza, Cincinnati, Ohio 45202 (US). KAUFMAN,  
Kathleen, Mary; One Procter & Gamble Plaza, Cincinnati,  
Ohio 45202 (US). DOYLE, Kevin, Lee; One Procter &  
Gamble Plaza, Cincinnati, Ohio 45202 (US). HOSSEIN-  
POUR, Dariush; One Procter & Gamble Plaza, Cincinnati,  
Ohio 45202 (US). ZHAO, Jean, Jianqun; One Procter &  
Gamble Plaza, Cincinnati, Ohio 45202 (US).(74) Agent: KREBS, Jay A.; c/o The Procter & Gamble Com-  
pany, Global IP Services, One Procter & Gamble Plaza, C9,  
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## (54) Title: METHOD OF FORMING A SILICONE LAYER



FIG. 1

(57) **Abstract:** A method of treating the hair including providing a concentrated hair care composition in a foam dispenser. The concentrated hair care composition includes one or more silicones, perfume, and less than 6% high melting point fatty compounds. The method also includes dispensing the concentrated hair care composition from the foam dispenser as a dosage of foam; applying the foam to the hair; and rinsing the foam from the hair. The foam has a density of from about 0.025 g/cm<sup>3</sup> to about 0.40 g/cm<sup>3</sup> when dispensed from the foam dispenser. The one or more silicones form a silicone layer that has a Targeted Deposition Index of from about 0.8 to about 1.6.



## METHOD OF FORMING A SILICONE LAYER

## FIELD OF THE INVENTION

Described herein is a method of forming a silicone layer with a concentrated hair care  
5 composition provided in a foam dispenser.

## BACKGROUND OF THE INVENTION

Today's hair conditioners almost universally comprise high levels of high melting point  
fatty compounds, the most common of which are C16 to C18 fatty alcohols. These high melting  
10 point fatty compounds are employed as structuring agents wherein they are combined with one or  
more surfactants and an aqueous carrier to form a gel network. The gel network provides a  
viscous and high yield point rheology which facilitates the dispensing of the conditioner from a  
bottle or tube and the subsequent distribution and spreading of the product through the hair by the  
consumer. The gel network structuring also enables incorporation of silicones, perfumes and oils  
15 in the form of an oil-in-water emulsion that is phase stable. These silicones and oils are intended  
to be deposited on the hair to provide the primary hair conditioning benefits including wet and  
dry combing friction reduction and hair manageability etc.

However, today's gel network hair conditioners lead to excessive co-deposits of the high  
melting point fatty compound on the hair over multiple cycles. Additionally, the deposited high  
20 melting point fatty compounds build-up on hair over multiple cycles and lead to significant waxy  
build-up on hair and hair weigh down. Indeed, one of the major consumer complaints with hair  
conditioners is waxy residue which makes hair look greasy or feel heavy. Many current gel  
network hair conditioners deposit up to 10 times more high melting point fatty compounds (fatty  
alcohols) than silicone or oil after approximately 10 treatment cycles in technical testing. While  
25 not being bound to theory, this is hypothesized to be due to the ~ 10x greater concentration of  
high melting point weight fatty compounds in the product relative to the silicone or oil. Such a  
high level of melting point fatty compounds (fatty alcohols) may be required to produce a shelf  
stable gel network with sufficient structuring for consumer acceptable viscosity and rheology.

Described herein is a concentrated hair care composition that enables new product  
30 opportunities and consumer benefits by addressing the current disadvantages associated with gel  
network conditioners. It has been found that concentrated and ultra-low viscosity hair  
conditioner compositions can be delivered to the hair in foamed form. These new concentrated  
silicone nanoemulsion compositions enable sufficient dosage from a foam delivery form while

also eliminating the need for high melting point fatty compounds or other “insoluble” structurants that can lead to significant co-deposits, build-up and weigh down of hair. The net result has been a step change improvement in silicone deposition versus today’s rinse-off products and an improvement in technical performance benefits from such a pure and transparent deposited  
5 silicone layer. These benefits include multicycle hair conditioning without hair weigh down, durable conditioning, reduced hair dye fade, and increased color vibrancy.

Nanoemulsion technology development is hindered by complex stability issues that emerge when droplet sizes are driven to the nanoscale. This may be especially problematic in the presence of higher levels of perfume oils which may be required for such a concentrated product.

10 The concentrated hair care composition described herein is therefor also focused on improved stability.

#### SUMMARY OF THE INVENTION

Described herein is a method of treating hair, the method comprising (a) providing a  
15 rinse-off concentrated hair care composition in a foam dispenser, wherein the concentrated hair care composition comprises (i) from about 3% to about 25% of one or more silicones, by weight of the concentrated hair care composition; (ii) less than 6% high melting point fatty compounds, by weight of the concentrated hair care composition; (iii) from about 60% to about 90% water, by weight of the concentrated hair care composition; and (iv) from about 0.5% to about 7% perfume,  
20 by weight of the concentrated hair care composition; wherein the concentrated hair care composition has a silicone to high melting point fatty compound weight ratio of from about 100:0 to about 50:50; wherein the concentrated hair care composition has a silicone to perfume weight ratio of from about 98:2 to about 50:50; (b) dispensing the concentrated hair care composition from the foam dispenser as a foam, wherein the foam has a density of from about 0.025 g/cm<sup>3</sup> to about 0.40 g/cm<sup>3</sup>; (c) applying the foam to the hair, wherein the one or more silicones are deposited onto the hair when applying the foam to the hair; and (d) rinsing the foam from the hair; wherein the one or more silicones forms a silicone layer on the hair; and wherein the silicone layer has a Targeted Deposition Index of from about 0.8 to about 1.6.

Also described herein is a foam dispenser comprising a concentrated hair care  
30 composition, wherein the concentrated hair care composition comprises (i) from about 3% to about 25% of one or more silicones, by weight of the concentrated hair care composition; (ii) less than 6% high melting point fatty compounds, by weight of the concentrated hair care composition; (iii) from about 60% to about 90% water, by weight of the concentrated hair care

composition; and (iv) from about 0.5% to about 7% perfume, by weight of the concentrated hair care composition; wherein the concentrated hair care composition is a rinse-off concentrated hair care composition; wherein the concentrated hair care composition has a silicone to high melting point fatty compound weight ratio of from about 100:0 to about 50:50; wherein the concentrated hair care composition has a silicone to perfume weight ratio of from about 98:2 to about 50:50; wherein the foam has a density of from about 0.025 g/cm<sup>3</sup> to about 0.40 g/cm<sup>3</sup>; wherein the rinse-off concentrated hair care composition deposits a silicone layer onto hair; wherein the silicone layer has a Targeted Deposition Index of from about 0.8 to about 1.6.

#### BRIEF DESCRIPTION OF THE DRAWINGS

While the specification concludes with claims, it is believed that the same will be better understood from the following description taken in conjunction with the accompanying drawings in which:

FIG. 1 is an SEM image of hair treated with a Pantene Clarifying Shampoo plus a Pantene Anti-breakage Conditioner;

FIG. 2 is an SEM image of hair treated with a Pantene Clarifying Shampoo plus the aerosol foam conditioner of Example 1 from Table 2;

FIG. 3 is an SEM image of hair treated with a Pantene Clarifying Shampoo plus the aerosol foam conditioner of Example 2 from Table 2;

FIG. 4 is an SEM image of hair treated with a Pantene Clarifying Shampoo plus the aerosol foam conditioner of Example 3 from Table 2;

FIG. 5 is an SEM image of hair treated with a Pantene Clarifying Shampoo plus the aerosol foam conditioner of Example 4 from Table 2;

FIG. 6 is an SEM image of hair treated with Foam Shampoo 1 from Table 1 plus the aerosol foam conditioner of Example 2 from Table 2; and

FIG. 7 is an SEM image of hair treated with Foam Shampoo 2 from Table 1 plus the aerosol foam conditioner of Example 2 from Table 2.

#### DETAILED DESCRIPTION OF THE INVENTION

While the specification concludes with claims particularly pointing out and distinctly claiming the invention, it is believed that the present invention will be better understood from the following description.

As used herein, the articles including "a" and "an" when used in a claim, are understood to mean one or more of what is claimed or described.

As used herein, "comprising" means that other steps and other ingredients which do not affect the end result can be added. This term encompasses the terms "consisting of" and "consisting essentially of".

As used herein, "mixtures" is meant to include a simple combination of materials and any compounds that may result from their combination.

As used herein, "molecular weight" or "M.Wt." refers to the weight average molecular weight unless otherwise stated.

As used herein, the terms "include," "includes," and "including," are meant to be non-limiting and are understood to mean "comprise," "comprises," and "comprising," respectively.

As used herein, the term "concentrated" means a hair care composition comprising from about 5% to about 22% of one or more silicones, by weight of the hair care composition.

As used herein, the term "nanoemulsion" means an oil-in-water (o/w) emulsion with an average particle size ranging from about 1 nm to about 100 nm. The particle size referred to herein is z-average measured by dynamic light scattering. The nanoemulsion described herein may be prepared by the following methods: (1) mechanically breaking down the emulsion droplet size; (2) spontaneously forming the emulsion (may be referred to as a microemulsion in the literature); and (3) using emulsion polymerization to achieve average particle size in the target range described herein.

All percentages, parts and ratios are based upon the total weight of the compositions of the present invention, unless otherwise specified. All such weights as they pertain to listed ingredients are based on the active level and, therefore, do not include carriers or by-products that may be included in commercially available materials.

Unless otherwise noted, all component or composition levels are in reference to the active portion of that component or composition, and are exclusive of impurities, for example, residual solvents or by-products, which may be present in commercially available sources of such components or compositions.

It should be understood that every maximum numerical limitation given throughout this specification includes every lower numerical limitation, as if such lower numerical limitations were expressly written herein. Every minimum numerical limitation given throughout this specification will include every higher numerical limitation, as if such higher numerical limitations were expressly written herein. Every numerical range given throughout this

specification will include every narrower numerical range that falls within such broader numerical range, as if such narrower numerical ranges were all expressly written herein.

### Hair Care Composition

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#### **A. Silicone Deposition Purity**

The method of treating hair comprises dispensing the concentrated hair care composition described herein from the aerosol foam dispenser as a dosage of foam. The foam may comprise a silicone deposition purity of from about 40% to about 100%, alternatively from about 50% to about 100%, alternatively from about 60% to about 100%, alternatively from about 70% to about 100%, alternatively from about 80% to about 100%, alternatively from about 85% to about 100%, alternatively from about 90% to about 100%, alternatively from about 70% to about 97%, alternatively from about 75% to about 97%, and alternatively from about 80% to about 97%, after applying the foam to the hair and rinsing the foam from the hair.

Deposition Purity is determined by the ratio of silicone deposited per weight of hair to the total deposition of other ingredients per weight of hair. Silicone is determined by either extraction or digestion of the hair followed by an analysis with a quantitative elemental technique such as ICP for total silicon and converting to silicone based on the % of silicon in the silicone by weight. The total deposition may be determined by the sum of separate deposition measurements or by a Single Inclusive Measurement of total deposition. The separate deposition measurements may include but are not limited to: fatty alcohols, EGDS, quaternized agents and silicone. Typically these measurements involve extracting the hair then separating the ingredients of interest with chromatography and quantifying with an external calibration based on test solution concentration. The Single Inclusive Measurement of total deposition is gravimetric. The hair is thoroughly extracted and the residue determined by weighing the dissolved residue in the extract after evaporating the solvent. This residue contains both deposited ingredients and naturally occurring extractable compounds from the hair (primarily lipids). The naturally occurring extractable compounds are quantified and subtracted from the total. These include: fatty acids, squalene, cholesterol, ceramides, wax esters, triglycerides and sterol esters. The method of quantitation is similar to the deposition measurements. Other supporting evidence of Deposition Purity may include spectroscopic or topography mapping of the hair surface.

## B. Silicones

The concentrated hair care composition may comprise from about 5% to about 20%, alternatively from about 8% to about 18%, and alternatively from about 10% to about 14% of one or more silicones, by weight of the concentrated hair care composition. In a further embodiment, the hair care composition may comprise from about 3% to about 25%, alternatively from about 4% to about 20%, alternatively from about 5% to about 15% of one or more silicones, and alternatively from about 6% to about 12% of one or more silicones by weight of the concentrated hair care composition. The particle size of the one or more silicones may be from about 1 nm to about 100 nm, alternatively from about 5 nm to about 80 nm, alternatively from about 10 nm to about 60 nm, and alternatively from about 12 nm to about 50 nm. In a further embodiment, the particle size of the one or more silicones may be from about 1 nm to about 500 nm, alternatively from about 5 nm to about 300 nm, alternatively from about 8 nm to about 200 nm, and alternatively from about 10 nm to about 100 nm.

The particle size of the one or more silicones may be measured by dynamic light scattering (DLS). A Malvern Zetasizer Nano ZEN3600 system ([www.malvern.com](http://www.malvern.com)) using He-Ne laser 633nm may be used for the measurement at 25°C. Prior low level centrifugation may be required on opaque formulas comprising larger insoluble structures (e.g., fatty alcohols) that would need to be isolated from the emulsion particles.

The autocorrelation function may be analyzed using the Zetasizer Software provided by Malvern Instruments, which determines the effective hydrodynamic radius, using the Stokes-Einstein equation:

$$D = \frac{k_B T}{6\pi\eta R}$$

wherein  $k_B$  is the Boltzmann Constant,  $T$  is the absolute temperature,  $\eta$  is the viscosity of the medium,  $D$  is the mean diffusion coefficient of the scattering species, and  $R$  is the hydrodynamic radius of particles.

Particle size (i.e. hydrodynamic radius) may be obtained by correlating the observed speckle pattern that arises due to Brownian motion and solving the Stokes-Einstein equation, which relates the particle size to the measured diffusion constant, as is known in the art.

Other methods known in the art may also be employed to measure particle size including cryo-SEM, cryo-TEM, and laser-diffraction methods..

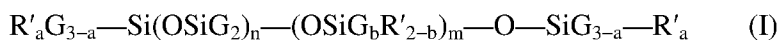
For each sample, 3 measurements may be made and Z-average values may be reported as the particle size.

In an embodiment, the one or more silicones may be in the form of a nanoemulsion. A nanoemulsion, as defined herein, is an emulsion wherein the particle size is below 100nm. The nanoemulsion may comprise any silicone suitable for application to the skin and/or hair. In an embodiment, from about 25% to about 100% of the one or more silicones is in the form of a nanoemulsion, in another embodiment from about 50% to about 100% of the one or more silicones is in the form of a nanoemulsion, and in another embodiment from about 75% to about 100% of the one or more silicones is in the form of a nanoemulsion, by weight of the hair care composition.

In an embodiment, the one or more silicones may include in their molecular structure polar functional groups such as Si-OH (present in dimethiconols), primary amines, secondary amines, tertiary amines, and quaternary ammonium salts. The one or more silicones may be selected from the group consisting of aminosilicones, pendant quaternary ammonium silicones, terminal quaternary ammonium silicones, amino polyalkylene oxide silicones, quaternary ammonium polyalkylene oxide silicones, and amino morpholino silicones.

The one or more silicones may comprise:

(a) at least one aminosilicone corresponding to formula (V):



in which:

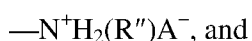
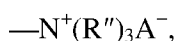
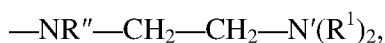
G is chosen from a hydrogen atom, a phenyl group, OH group, and C<sub>1</sub>-C<sub>8</sub> alkyl groups, for example methyl,

a is an integer ranging from 0 to 3, and in one embodiment a is 0,

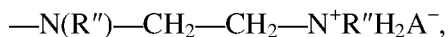
b is chosen from 0 and 1, and in one embodiment b is 1,

m and n are numbers such that the sum (n+m) can range for example from 1 to 2 000, such as for example from 50 to 150, wherein n can be for example chosen from numbers ranging from 0 to 1 999, such as for example from 49 to 149, and wherein m can be chosen from numbers ranging for example from 1 to 2 000, such as for example from 1 to 10;

R' is a monovalent group of formula —C<sub>q</sub>H<sub>2q</sub>L in which q is a number from 2 to 8 and L is an optionally quaternized amine group chosen from the groups:





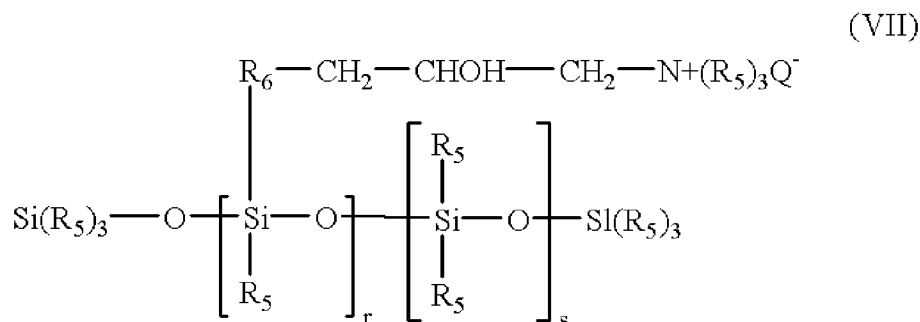


in which R'' can be chosen from a hydrogen atom, phenyl groups, benzyl groups, and saturated monovalent hydrocarbon-based groups, such as for example an alkyl group comprising from 1 to 20 carbon atoms, and A<sup>−</sup> is chosen from halide ions such as, for example, fluoride, chloride, bromide and iodide.

In an embodiment, the one or more silicones may include those corresponding to formula (1) wherein a = 0, G=methyl, m and n are numbers such that the sum (n+m) can range for example from 1 to 2 000, such as for example from 50 to 150, wherein n can be for example chosen from numbers ranging from 0 to 1 999, such as for example from 49 to 149, and wherein m can be chosen from numbers ranging for example from 1 to 2 000, such as for example from 1 to 10; and L is —N(CH<sub>3</sub>)<sub>2</sub> or —NH<sub>2</sub>, alternatively —NH<sub>2</sub>.

Additional said at least one aminosilicone of the invention include:

(b) pendant quaternary ammonium silicones of formula (VII):



in which:

R<sub>5</sub> is chosen from monovalent hydrocarbon-based groups comprising from 1 to 18 carbon atoms, such as C<sub>1</sub>-C<sub>18</sub> alkyl groups and C<sub>2</sub>-C<sub>18</sub> alkenyl groups, for example methyl;

R<sub>6</sub> is chosen from divalent hydrocarbon-based groups, such as divalent C<sub>1</sub>-C<sub>18</sub> alkylene groups and divalent C<sub>1</sub>-C<sub>18</sub> alkyleneoxy groups, for example C<sub>1</sub>-C<sub>8</sub> alkyleneoxy groups, wherein said R<sub>6</sub> is bonded to the Si by way of a SiC bond;

Q<sup>−</sup> is an anion that can be for example chosen from halide ions, such as chloride, and organic acid salts (such as acetate);

r is an average statistical value ranging from 2 to 20, such as from 2 to 8;

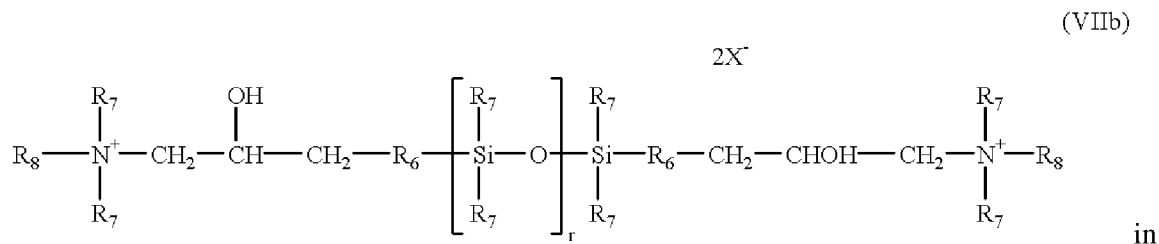
s is an average statistical value ranging from 20 to 200, such as from 20 to 50.

Such aminosilicones are described more particularly in U.S. Pat. No. 4,185,087, the disclosure of which is incorporated by reference herein.

A silicone which falls within this class is the silicone sold by the company Union Carbide under the name “Ucar Silicone ALE 56”.

Further examples of said at least one aminosilicone include:

c) quaternary ammonium silicones of formula (VIIb):



which:

- 5 groups  $\text{R}_7$ , which may be identical or different, are each chosen from monovalent hydrocarbon-based groups comprising from 1 to 18 carbon atoms, such as  $\text{C}_1$ - $\text{C}_{18}$  alkyl groups, for example methyl,  $\text{C}_2$ - $\text{C}_{18}$  alkenyl groups, and rings comprising 5 or 6 carbon atoms;

$\text{R}_6$  is chosen from divalent hydrocarbon-based groups, such as divalent  $\text{C}_1$ - $\text{C}_{18}$  alkylene groups and divalent  $\text{C}_1$ - $\text{C}_{18}$  alkyleneoxy, for example  $\text{C}_1$ - $\text{C}_8$ , group connected to the Si by a SiC bond;

- 10  $\text{R}_8$ , which may be identical or different, represent a hydrogen atom, a monovalent hydrocarbon-based group comprising from 1 to 18 carbon atoms, and in particular a  $\text{C}_1$ - $\text{C}_{18}$  alkyl group, a  $\text{C}_2$ - $\text{C}_{18}$  alkenyl group or a group  $-\text{R}_6-\text{NHCOR}_7$ ;

$\text{X}^-$  is an anion such as a halide ion, in particular chloride, or an organic acid salt (acetate, etc.);

$r$  represents an average statistical value from 2 to 200 and in particular from 5 to 100.

- 15 Such silicones are described, for example, in application EP-A-0 530 974, the disclosure of which is incorporated by reference herein.

Silicones falling within this class are the silicones sold by the company Goldschmidt under the names Abil Quat 3270, Abil Quat 3272 and Abil Quat 3474.

Further examples of said at least one aminosilicone include:

- 20 d) quaternary ammonium and polyalkylene oxide silicones

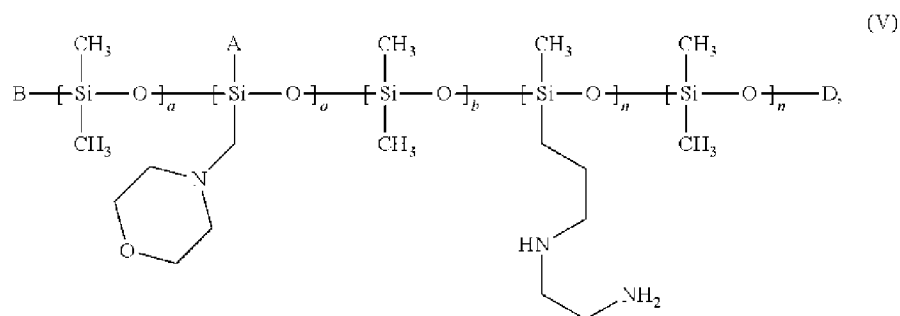
wherein the quaternary nitrogen groups are located in the polysiloxane backbone, at the termini, or both.

Such silicones are described in PCT Publication No. WO 2002/010257, the disclosure of which is incorporated by reference herein.

- 25 Silicones falling within this class are the silicones sold by the company Momentive under the names, i.e., Silsoft Q.

(e) Aminofunctional silicones having morpholino groups of formula (V):

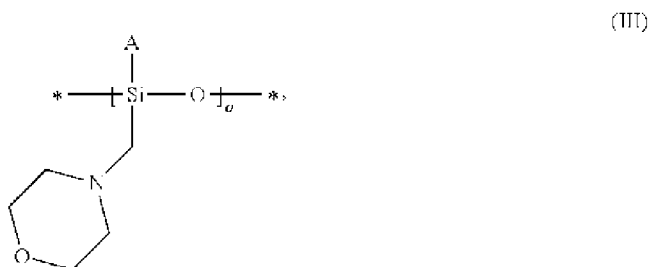
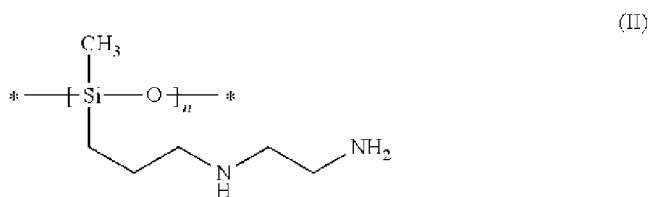
10



in which

A denotes a structural unit (I), (II), or (III) bound via —O—

5



or an oligomeric or polymeric residue, bound via —O—, containing structural units of formulas (I), (II), or (III), or half of a connecting oxygen atom to a structural unit (III), or denotes —OH,

10

\* denotes a bond to one of the structural units (I), (II), or (III), or denotes a terminal group B (Si-bound) or D (O-bound),

B denotes an —OH, —O—Si(CH<sub>3</sub>)<sub>3</sub>, —O—Si(CH<sub>3</sub>)<sub>2</sub>OH, —O—Si(CH<sub>3</sub>)<sub>2</sub>OCH<sub>3</sub> group,

D denotes an —H, —Si(CH<sub>3</sub>)<sub>3</sub>, —Si(CH<sub>3</sub>)<sub>2</sub>OH, —Si(CH<sub>3</sub>)<sub>2</sub>OCH<sub>3</sub> group,

15

a, b, and c denote integers between 0 and 1000, with the provision that a+b+c>0,

m, n, and o denote integers between 1 and 1000.

Aminofunctional silicones of this kind bear the INCI name: Amodimethicone/Morpholinomethyl Silsesquioxane Copolymer. A particularly suitable amodimethicone is the product having the commercial name Wacker Belsil® ADM 8301E.

Examples of such silicones are available from the following suppliers:

5                   *offered by the company Dow Corning:*

Fluids: 2-8566, AP 6087, AP 6088, DC 8040 Fluid, fluid 8822A DC, DC 8803 & 8813 polymer, 7-6030, AP-8104, AP 8201;

Emulsions: CE-8170 AF Micro Emulsion, 2-8177, 2-8194 Microemulsion, 9224 Emulsion, 939, 949, 959, DC 5-7113 Quat Microemulsion, DC 5-7070 Emulsion, DC CE-8810, CE 8401 Emulsion, CE 1619, Dow Corning Toray SS-3551, Dow Corning Toray SS-3552;

*offered by the company Wacker:*

Wacker Belsil ADM 652, ADM 656, 1100, 1600, 1650 (fluids) ADM 6060 (linear amodimethicone) emulsion; ADM 6057 E (branched amodimethicone) emulsion; ADM 8020 VP (micro emulsion); SLM 28040 (micro emulsion);

*offered by the Company Momentive:*

Silsoft 331, SF1708, SME 253 & 254 (emulsion), SM2125 (emulsion), SM 2658 (emulsion), Silsoft Q (emulsion)

20                   *offered by the company Shin-Etsu:*

KF-889, KF-867S, KF-8004, X-52-2265 (emulsion);

*offered by the Company Siltech Silicones:*

Siltech E-2145, E-Siltech 2145-35;

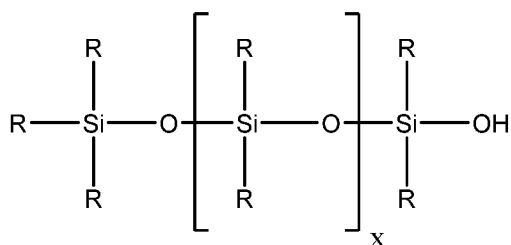
*offered by the company Evonik Industries:*

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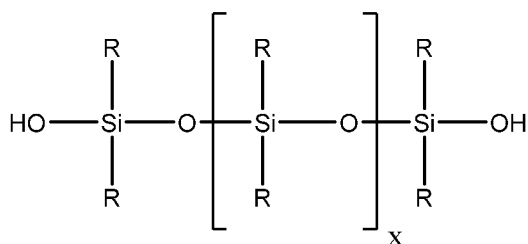
Some non-limiting examples of aminosilicones include the compounds having the following INCI names: Silicone Quaternium-1, Silicone Quaternium-2, Silicone Quaternium-3, Silicone Quaternium-4, Silicone Quaternium-5, Silicone Quaternium-6, Silicone Quaternium-7, Silicone Quaternium-8, Silicone Quaternium-9, Silicone Quaternium-10, Silicone Quaternium-11, Silicone Quaternium-12, Silicone Quaternium-15, Silicone Quaternium-16, Silicone Quaternium-17, Silicone Quaternium-18, Silicone Quaternium-20, Silicone Quaternium-21, Silicone Quaternium-22, Quaternium-80, as well as Silicone Quaternium-2 Panthenol Succinate and Silicone Quaternium-16/Glycidyl Dimethicone Crosspolymer.

In an embodiment, the aminosilicones can be supplied in the form of a nanoemulsion and include MEM 9049, MEM 8177, MEM 0959, MEM 8194, SME 253, and Silsoft Q.

In an embodiment, the one or more silicones may include dimethicones, and/or dimethiconols. The dimethiconols are hydroxyl terminated dimethylsilicones represented by the  
5 general chemical formulas



and



wherein R is an alkyl group (R may be methyl or ethyl) and x is an integer up to about 500,  
15 chosen to achieve the desired molecular weight. Commercial dimethiconols typically are sold as mixtures with dimethicone or cyclomethicone (e.g., Dow Coming® 1401, 1402, and 1403 fluids).

### C. Nonionic Emulsifiers

The concentrated hair care composition may comprise from about 3% to about 20%,  
20 alternatively from about 5% to about 15%, and alternatively from about 7.5% to about 12% of a nonionic emulsifier, by weight of the concentrated hair care composition. In an embodiment, the concentrated hair care composition may comprise from about 0% to about 20%, alternatively from about 0.01% to about 20%, alternatively from about 1% to about 15%, alternatively from about 2% to about 12%, alternatively from about 3% to about 10%, and alternatively from about  
25 4% to about 8% of a nonionic emulsifier, by weight of the concentrated hair care composition.

Nonionic emulsifiers may be broadly defined as including compounds containing an alkylene oxide groups (hydrophilic in nature) with a hydrophobic compound, which may be aliphatic or alkyl aromatic in nature. Examples of nonionic emulsifiers include:

1. Alcohol ethoxylates which are condensation products of aliphatic alcohols having from about 8 to about 18 carbon atoms, in either straight chain or branched chain configuration, with from about 2 to about 35 moles of ethylene oxide, e.g., a coconut alcohol ethylene oxide condensate having from about 2 to about 30 moles of ethylene oxide per mole of coconut alcohol, the coconut alcohol fraction having from about 10 to about 14 carbon atom.

2. The polyethylene oxide condensates of alkyl phenols, e.g., the condensation products of the alkyl phenols having an alkyl group containing from about 6 to about 20 carbon atoms in either a straight chain or branched chain configuration, with ethylene oxide, the said ethylene oxide being present in amounts equal to from about 3 to about 60 moles of ethylene oxide per mole of alkyl phenol.

3. Those derived from the condensation of ethylene oxide with the product resulting from the reaction of propylene oxide and ethylene diamine products.

4. Long chain tertiary amine oxides such as those corresponding to the following general formula:  $R_1 R_2 R_3 N \rightarrow O$  wherein  $R_1$  contains an alkyl, alkenyl or monohydroxy alkyl radical of from about 8 to about 18 carbon atoms, from 0 to about 10 ethylene oxide moieties, and from 0 to about 1 glyceryl moiety, and  $R_2$  and  $R_3$  contain from about 1 to about 3 carbon atoms and from 0 to about 1 hydroxy group, e.g., methyl, ethyl, propyl, hydroxyethyl, or hydroxypropyl radicals (the arrow in the formula represents a semipolar bond).

5. Long chain tertiary phosphine oxides corresponding to the following general formula:  $RR'R''P \rightarrow O$  wherein  $R$  contains an alkyl, alkenyl or monohydroxyalkyl radical ranging from about 8 to about 18 carbon atoms in chain length, from 0 to about 10 ethylene oxide moieties and from 0 to about 1 glyceryl moiety and  $R'$  and  $R''$  are each alkyl or monohydroxyalkyl groups containing from about 1 to about 3 carbon atoms. The arrow in the formula represents a semipolar bond.

6. Long chain dialkyl sulfoxides containing one short chain alkyl or hydroxy alkyl radical of from about 1 to about 3 carbon atoms (usually methyl) and one long hydrophobic chain which include alkyl, alkenyl, hydroxy alkyl, or keto alkyl radicals containing from about 8 to about 20 carbon atoms, from 0 to about 10 ethylene oxide moieties and from 0 to about 1 glyceryl moiety.

7. Polysorbates, e.g., sucrose esters of fatty acids. Such materials are described in U.S. Patent 3,480,616, e.g., sucrose cocoate (a mixture of sucrose esters of a coconut acid, consisting

primarily of monoesters, and sold under the trade names GRILLOTEN LSE 87K from RITA, and CRODESTA SL-40 from Croda).

8. Alkyl polysaccharide nonionic emulsifiers are disclosed in U.S. Patent 4,565,647, Llenado, issued January 21, 1986, having a hydrophobic group containing from about 6 to about 30 carbon atoms, alternatively from about 10 to about 16 carbon atoms and a polysaccharide, e.g., a polyglycoside, hydrophilic group. The polysaccharide can contain from about 1.0 to about 10, alternatively from about 1.3 to about 3, and alternatively from about 1.3 to about 2.7 saccharide units. Any reducing saccharide containing 5 or 6 carbon atoms can be used, e.g., glucose, galactose and galactosyl moieties can be substituted for the glucosyl moieties. (Optionally the hydrophobic group is attached at the 2-,3-, 4-, etc. positions thus giving a glucose or galactose as opposed to a glucoside or galactoside.) The intersaccharide bonds can be, e.g., between the one position of the additional saccharide units and the 2-, 3-, 4-, and/or 6-positions on the preceding saccharide units. Optionally there can be a polyalkyleneoxide chain joining the hydrophobic moiety and the polysaccharide moiety. The alkyl group may contain up to about 3 hydroxy groups and/or the polyalkyleneoxide chain can contain up to about 10, alternatively less than 5, alkylene moieties. Suitable alkyl polysaccharides are octyl, nonyldecyl, undecyldodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl, and octadecyl, di-, tri-, tetra-, penta-, and hexagluco-sides, galactosides, lactosides, glucoses, fructosides, fructoses and/or galactoses.

9. Polyethylene glycol (PEG) glyceryl fatty esters, as depicted by the formula  $RC(O)OCH_2CH(OH)CH_2(OCH_2CH_2)_nOH$  wherein n is from about 5 to about 200, alternatively from about 20 to about 100, alternatively from about 30 to about 85, and RC(O)- is an ester wherein R comprises an aliphatic radical having from about 7 to 19 carbon atoms, alternatively from about 9 to 17 carbon atoms, alternatively from about 11 to 17 carbon atoms, alternatively from about 11 to 14 carbon atoms. In an embodiment, the combinations of n may be from about 20 to about 100, with C12 -C18, alternatively C12 -C15 fatty esters, for minimized adverse effect on foaming.

In an embodiment, the nonionic emulsifier may be a silicone emulsifier. A wide variety of silicone emulsifiers may be useful herein. These silicone emulsifiers are typically organically modified siloxanes, also known to those skilled in the art as silicone surfactants. Useful silicone emulsifiers include dimethicone copolyols. These materials are polydimethyl siloxanes which have been modified to include polyether side chains such as polyethylene oxide chains, polypropylene oxide chains, mixtures of these chains, and polyether chains containing moieties derived from both ethylene oxide and propylene oxide. Other examples include alkyl-modified

dimethicone copolyols, i.e., compounds which contain C2-C30 pendant side chains. Still other useful dimethicone copolyols include materials having various cationic, anionic, amphoteric, and zwitterionic pendant moieties.

In an embodiment, the nonionic emulsifier may have a hydrocarbon chain length of from about 16 to about 20 carbon atoms and from about 20 to about 25 moles of ethoxylate.

In an embodiment, the nonionic emulsifier may have a hydrocarbon chain length of from about 19 to about 11, alternatively from about 9 to about 11 carbon atoms, and from about 2 to about 4 moles of ethoxylate.

In an embodiment, the nonionic emulsifier may comprise a combination of (a) a nonionic emulsifier having a hydrocarbon chain that is branched, has a length of from about 11 to about 15 carbon atoms, and has from about 5 to about 9 moles of ethoxylate; and (b) a nonionic emulsifier having a hydrocarbon chain that has a length of from about 11 to about 13 carbon atoms and has from about 9 to about 12 moles of ethoxylate.

The nanoemulsions used in this invention may be prepared by two different methods: (1) mechanical, and (2) emulsion polymerization.

The first method of preparing the nanoemulsion is the mechanical method in which the nanoemulsion is prepared via the following steps: (1) a primary surfactant is dissolved in water, (2) a silicone is added, and a two-phase mixture is formed, (3) with simple mixing, a co-surfactant is slowly added to the two-phase mixture, until a clear isotropic microemulsion of a siloxane-in-water is formed.

The second method of preparing the nanoemulsion is by emulsion polymerization. Emulsion polymerization methods for making nanoemulsions of polymers involve starting with polymer precursors, i.e., monomers, or reactive oligomers, which are immiscible in water; a surfactant to stabilize polymer precursor droplets in water; and a water soluble polymerization catalyst. Typically, the catalyst is a strong mineral acid such as hydrochloric acid, or a strong alkaline catalyst such as sodium hydroxide. These components are added to water, the mixture is stirred, and polymerization is allowed to advance until the reaction is complete, or the desired degree of polymerization (DP) is reached, and an emulsion of the polymer is formed.

#### **D. Perfume**

The concentrated hair care composition may comprise from about 0.5% to about 7%, alternatively from about 1% to about 6%, and alternatively from about 2% to about 5% perfume, by weight of the concentrated hair care composition.



In an embodiment, the concentrated hair care composition may have a silicone to perfume weight ratio of from about 98:2 to about 50:50, alternatively from about 95:5 to about 50:50, alternatively from about 90:10 to about 60:40, and alternatively from about 85:15 to about 70:30.

Examples of suitable perfumes may be provided in the CTFA (Cosmetic, Toiletry and  
5 Fragrance Association) 1992 International Buyers Guide, published by CFTA Publications and OPD 1993 Chemicals Buyers Directory 80th Annual Edition, published by Schnell Publishing Co. A plurality of perfume components may be present in the concentrated hair care composition.

#### 10 E. High Melting Point Fatty Compounds

The concentrated hair care composition may comprise less than 10% high melting point fatty compounds, alternatively less than 8% high melting point fatty compounds, alternatively less than 6% high melting point fatty compounds, alternatively less than 5% high melting point fatty compounds, alternatively less than 3% high melting point fatty compound, alternatively may  
15 be substantially free of high melting point fatty compounds, and alternatively may comprise 0% high melting point fatty compounds, by weight of the concentrated hair care composition. In an embodiment, the hair care composition may comprise from about 0% to about 8% fatty alcohols, alternatively from about 0.5% to about 6%, alternatively from about 1.0% to about 4%, and alternatively from about 1.5% to about 3.0% fatty alcohols, by weight of the concentrated hair  
20 care composition. The concentrated hair care composition may have a silicone to high melting point fatty compounds weight ratio of from about 100:0 to about 40:60, alternatively from about 100:0 to about 50:50, alternatively from about 100:0 to about 60:40, alternatively from about 100:0 to about 65:35, alternatively from about 100:0 to about 75:25, alternatively from about 100:0 to about 80:20, alternatively from about 100:0 to about 85:15, alternatively from about 95:5  
25 to about 65:35, alternatively from about 95:5 to about 75:25, alternatively from about 95:5 to about 80:20, and alternatively from about 80:20 to about 65:35. In an embodiment the concentrated hair care composition may have a silicone to high melting point fatty compounds weight ratio of from about 100:0 to about 70:30.

The high melting point fatty compounds may have a melting point of about 25°C or  
30 higher, and can be chosen from fatty alcohols, fatty acids, fatty alcohol derivatives, fatty acid derivatives, and mixtures thereof. It is understood by the artisan that the compounds disclosed in this section of the specification can in some instances fall into more than one classification, e.g., some fatty alcohol derivatives can also be classified as fatty acid derivatives. However, a given

classification is not intended to be a limitation on that particular compound, but is done so for convenience of classification and nomenclature. Further, depending on the number and position of double bonds, and length and position of the branches, certain compounds having certain required carbon atoms may have a melting point of less than about 25°C. Such compounds of low melting point are not intended to be included in this section. Nonlimiting examples of the high melting point compounds are found in *International Cosmetic Ingredient Dictionary*, Fifth Edition, 1993, and *CTFA Cosmetic Ingredient Handbook*, Second Edition, 1992.

The fatty alcohols described herein can be those having from about 14 to about 30 carbon atoms, alternatively from about 16 to about 22 carbon atoms. These fatty alcohols are saturated and can be straight or branched chain alcohols. Nonlimiting examples of fatty alcohols include cetyl alcohol, stearyl alcohol, behenyl alcohol, and mixtures thereof.

The fatty acids useful herein are those having from about 10 to about 30 carbon atoms, alternatively from about 12 to about 22 carbon atoms, and alternatively from about 16 to about 22 carbon atoms. These fatty acids are saturated and can be straight or branched chain acids. Also included are diacids, triacids, and other multiple acids. Also included herein are salts of these fatty acids. Nonlimiting examples of fatty acids include lauric acid, palmitic acid, stearic acid, behenic acid, sebacic acid, and mixtures thereof.

The fatty alcohol derivatives and fatty acid derivatives useful herein include alkyl ethers of fatty alcohols, alkoxylated fatty alcohols, alkyl ethers of alkoxylated fatty alcohols, esters of fatty alcohols, fatty acid esters of compounds having esterifiable hydroxy groups, hydroxy-substituted fatty acids, and mixtures thereof. Nonlimiting examples of fatty alcohol derivatives and fatty acid derivatives include materials such as methyl stearyl ether; the ceteth series of compounds such as ceteth-1 through ceteth-45, which are ethylene glycol ethers of cetyl alcohol, wherein the numeric designation indicates the number of ethylene glycol moieties present; the steareth series of compounds such as steareth- 1 through steareth- 10, which are ethylene glycol ethers of steareth alcohol, wherein the numeric designation indicates the number of ethylene glycol moieties present; cetareth 1 through cetareth-10, which are the ethylene glycol ethers of cetareth alcohol, i.e., a mixture of fatty alcohols containing predominantly cetyl and stearyl alcohol, wherein the numeric designation indicates the number of ethylene glycol moieties present; C16 -C30 alkyl ethers of the ceteth, steareth, and cetareth compounds just described; polyoxyethylene ethers of behenyl alcohol; ethyl stearate, cetyl stearate, cetyl palmitate, stearyl stearate, myristyl myristate, polyoxyethylene cetyl ether stearate, polyoxyethylene stearyl ether stearate, polyoxyethylene lauryl ether stearate, ethyleneglycol monostearate, polyoxyethylene

monostearate, polyoxyethylene distearate, propyleneglycol monostearate, propyleneglycol distearate, trimethylolpropane distearate, sorbitan stearate, polyglyceryl stearate, glyceryl monostearate, glyceryl distearate, glyceryl tristearate, and mixtures thereof.

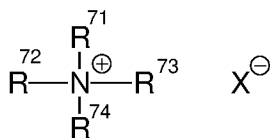
In an embodiment, the fatty compound may be a single high melting point compound of high purity. Single compounds of pure fatty alcohols selected may be selected from the group consisting of pure cetyl alcohol, stearyl alcohol, and behenyl alcohol. By "pure" herein, what is meant is that the compound has a purity of at least about 90%, alternatively at least about 95%.

Commercially available high melting point fatty compounds described herein include: cetyl alcohol, stearyl alcohol, and behenyl alcohol having trade names KONOL series available from Shin Nihon Rika (Osaka, Japan), and NAA series available from NOF (Tokyo, Japan); pure behenyl alcohol having trade name 1-DOCOSANOL available from WAKO (Osaka, Japan), various fatty acids having trade names NEO-FAT available from Akzo (Chicago, Illinois USA), HYSTRENE available from Witco Corp. (Dublin, Ohio USA), and DERMA available from Vevy (Genova, Italy).

#### F. Cationic Surfactants

In an embodiment, the concentrated hair care composition may comprise 0%, alternatively from about 0.25% to about 5%, alternatively from about 0.5% to about 4%, and alternatively from about 1% to about 3% cationic surfactants, by weight of the concentrated hair care composition.

The cationic surfactant may be a mono-long alkyl quaternized ammonium salt having the formula (XIII) [from WO2013148778]:



(XIII)

wherein one of  $\text{R}^{71}$ ,  $\text{R}^{72}$ ,  $\text{R}^{73}$  and  $\text{R}^{74}$  selected from an aliphatic group of from about 14 to about 30 carbon atoms or an aromatic, alkoxy, polyoxyalkylene, alkylamido, hydroxyalkyl, aryl or alkylaryl group having up to about 30 carbon atoms; the remainder of  $\text{R}^{71}$ ,  $\text{R}^{72}$ ,  $\text{R}^{73}$  and  $\text{R}^{74}$  are independently selected from an aliphatic group of from about 1 to about 8 carbon atoms or an aromatic, alkoxy, polyoxyalkylene, alkylamido, hydroxyalkyl, aryl or alkylaryl group having up to about 8 carbon atoms; and X is a salt-forming anion such as those selected from halogen, (e.g.,

chloride, bromide), acetate, citrate, lactate, glycolate, phosphate, nitrate, sulfonate, sulfate, alkylsulfate, glutamate, and alkyl sulfonate radicals. The aliphatic groups can contain, in addition to carbon and hydrogen atoms, ether linkages, and other groups such as amino groups. The longer chain aliphatic groups, e.g., those of about 16 carbons, or higher, can be saturated or unsaturated.

5 In an embodiment, one of  $R^{71}$ ,  $R^{72}$ ,  $R^{73}$  and  $R^{74}$  is selected from an alkyl group of from about 14 to about 30 carbon atoms, alternatively from about 16 to about 22 carbon atoms, alternatively from about 16 to about 18 carbon atoms; the remainder of  $R^{71}$ ,  $R^{72}$ ,  $R^{73}$ , and  $R^{74}$  are independently selected from the group consisting of  $CH_3$ ,  $C_2H_5$ ,  $C_2H_4OH$ ,  $CH_2C_5H_5$ , and mixtures thereof; and (X) is selected from the group consisting of Cl, Br,  $CH_3OSO_3$ , and mixtures  
10 thereof. It is believed that such mono-long alkyl quaternized ammonium salts can provide improved slippery and slick feel on wet hair.

Nonlimiting examples of such mono-long alkyl quaternized ammonium salt cationic surfactants include: behenyl trimethyl ammonium chloride available, for example, with trade name Genamine KDMP from Clariant, with trade name INCROQUAT TMC-80 from Croda and  
15 ECONOL TM22 from Sanyo Kasei; stearyl trimethyl ammonium chloride available, for example, with trade name CA-2450 from Nikko Chemicals; cetyl trimethyl ammonium chloride available, for example, with trade name CA-2350 from Nikko Chemicals; behenyltrimethylammonium methyl sulfate, available from FeiXiang; hydrogenated tallow alkyl trimethyl ammonium chloride; stearyl dimethyl benzyl ammonium chloride; and stearyl amidopropyl dimethyl benzyl  
20 ammonium chloride.

Among them, more preferred cationic surfactants are those having a shorter alkyl group, i.e.,  $C_{16}$  alkyl group. Such cationic surfactant includes, for example, cetyl trimethyl ammonium chloride. It is believed that cationic surfactants having a shorter alkyl group are advantageous for concentrated hair care silicone nanoemulsion compositions of the present invention comprising a  
25 cationic surfactant and with improved shelf stability.

In an embodiment, the concentrated hair care composition has a silicone to (high melting point fatty compound plus cationic surfactant) weight ratio of from about 0.30 to about 10, alternatively from about 0.40 to about 8, alternatively from about 0.50 to about 7, alternatively from about 0.60 to about 6, and alternatively from about 0.70 to about 5.

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### G. Water Miscible Solvents

The concentrated hair care compositions described herein may comprise from about 0.1% to about 25%, alternatively from about 0.1% to about 20%, and alternatively from about 0.1% to

about 15% of a water miscible solvent, by weight of the concentrated hair care composition. Non-limiting examples of suitable water miscible solvents include polyols, copolyols, polycarboxylic acids, polyesters and alcohols.

Examples of useful polyols include, but are not limited to, glycerin, diglycerin, propylene glycol, ethylene glycol, butylene glycol, pentylene glycol, 1,3-butylene glycol, cyclohexane dimethanol, hexane diol, polyethylene glycol (200-600), sugar alcohols such as sorbitol, manitol, lactitol and other mono- and polyhydric low molecular weight alcohols (e.g., C<sub>2</sub>-C<sub>8</sub> alcohols); mono di- and oligo-saccharides such as fructose, glucose, sucrose, maltose, lactose, and high fructose corn syrup solids and ascorbic acid.

Examples of polycarboxylic acids include, but are not limited to citric acid, maleic acid, succinic acid, polyacrylic acid, and polymaleic acid.

Examples of suitable polyesters include, but are not limited to, glycerol triacetate, acetylated-monoglyceride, diethyl phthalate, triethyl citrate, tributyl citrate, acetyl triethyl citrate, acetyl tributyl citrate.

Examples of suitable dimethicone copolyols include, but are not limited to, PEG-12 dimethicone, PEG/PPG-18/18 dimethicone, and PPG-12 dimethicone.

Examples of suitable alcohols include, but are not limited to ethanol, n-propanol, isopropanol, n-butanol, sec-butanol, tert-butanol, n-hexanol and cyclohexanol.

Other suitable water miscible solvents include, but are not limited to, alkyl and allyl phthalates; naphthalates; lactates (e.g., sodium, ammonium and potassium salts); sorbeth-30; urea; lactic acid; sodium pyrrolidone carboxylic acid (PCA); sodium hyaluronate or hyaluronic acid; soluble collagen; modified protein; monosodium L-glutamate; alpha & beta hydroxyl acids such as glycolic acid, lactic acid, citric acid, maleic acid and salicylic acid; glyceryl polymethacrylate; polymeric plasticizers such as polyquaterniums; proteins and amino acids such as glutamic acid, aspartic acid, and lysine; hydrogen starch hydrolysates; other low molecular weight esters (e.g., esters of C<sub>2</sub>-C<sub>10</sub> alcohols and acids); and any other water soluble plasticizer known to one skilled in the art of the foods and plastics industries; and mixtures thereof.

In an embodiment, the water miscible solvents may be selected from the group consisting of glycerin, propylene glycol, dipropylene glycol, and mixtures thereof. EP 0283165 B1 discloses other suitable water miscible solvents, including glycerol derivatives such as propoxylated glycerol.

## H. Viscosity Modifiers

The concentrated hair care composition described herein may comprise from about 0.1% to about 2%, alternatively from about 0.1% to about 1%, and alternatively from about 0.1% to about 0.5% of a viscosity modifier, by weight of the concentrated hair care composition. Non-limiting examples of suitable viscosity modifiers include water soluble polymers and cationic water soluble polymers.

Examples of water soluble polymers include, but are not limited to (1) vegetable based polymers such as gum Arabic, tragacanth gum, galactan, guar gum, carob gum, karaya gum, carrageenan, pectin, agar, quince seed, algal colloid, starch (rice, corn, potato, or wheat), and glycyrrhizinic acid; (2) microorganism-based polymers such as xanthan gum, dextran, succinoglucan, and pullulan; and (3) animal-based polymers such as collagen, casein, albumin, and gelatin. Examples of semi-synthetic water-soluble polymers include (1) starch-based polymers such as carboxymethyl starch and methylhydroxypropyl starch; (2) cellulose-based polymers such as methylcellulose, nitrocellulose, ethylcellulose, methylhydroxypropylcellulose, hydroxyethylcellulose, sodium cellulose sulfate, hydroxypropylcellulose, sodium carboxymethylcellulose (CMC), crystalline cellulose, and cellulose powder; and (3) alginate-based polymers such as sodium alginate and propylene glycol alginate. Examples of synthetic water-soluble polymers include (1) vinyl-based polymers such as polyvinyl alcohol, polyvinyl methyl ether- based polymer, polyvinylpyrrolidone, and carboxyvinyl polymer (CARBOPOL 940, CARBOPOL 941; (2) polyoxyethylene-based polymers such as polyethylene glycol 20,000, polyethylene glycol 6,000, and polyethylene glycol 4,000; (3) copolymer-based polymers such as a copolymer of polyoxyethylene and polyoxypropylene, and PEG/PPG methyl ether; (4) acryl-based polymers such as poly(sodium acrylate), poly(ethyl acrylate), polyacrylamide, polyethylene imines, and cationic polymers. The water-swellaable clay minerals are nonionic water-soluble polymers and correspond to one type of colloid-containing aluminum silicate having a triple layer structure. More particular, as examples thereof, mention may be made of bentonite, montmorillonite, beidellite, nontronite, saponite, hectorite, aluminum magnesium silicate, and silicic anhydride.

Examples of cationic water soluble polymers include, but are not limited to (1) quaternary nitrogen-modified polysaccharides such as cation-modified cellulose, cation-modified hydroxyethylcellulose, cation-modified guar gum, cation-modified locust bean gum, and cation-modified starch; (2) dimethyldiallylammonium chloride derivatives such as a copolymer of dimethyldiallylammonium chloride and acrylamide, and poly(dimethylmethylenepiperidinium

chloride); (3) vinylpyrrolidone derivatives such as a salt of a copolymer of vinylpyrrolidone and dimethylaminoethyl methacrylic acid, a copolymer of vinylpyrrolidone and methacrylamide propyltrimethylammonium chloride, and a copolymer of vinylpyrrolidone and methylvinylimidazolium chloride; and (4) methacrylic acid derivatives such as a copolymer of methacryloylethyldimethylbetaine, methacryloylethyl trimethylammonium chloride and 2-hydroxyethyl methacrylate, a copolymer of methacryloylethyldimethylbetaine, and methacryloylethyl trimethylammonium chloride and methoxy polyethylene glycol methacrylate.

### **I. Viscosity**

The concentrated hair care composition described herein may have a liquid phase viscosity of from about 1 centipoise to about 2,500 centipoise, alternatively from about 5 centipoise to about 2,000 centipoise, alternatively from about 10 centipoise to about 1,500 centipoise, and alternatively from about 15 centipoise to about 1,000 centipoise. In an embodiment, the concentrated hair care composition described herein may have a liquid phase viscosity of from about 1 centipoise to about 15,000 centipoise, alternatively from about 1 centipoise to about 8,000 centipoise, alternatively from about 5 centipoise to about 5,000 centipoise, alternatively from about 10 centipoise to about 2,500 centipoise, alternatively from about 15 centipoise to about 1,500 centipoise, and alternatively from about 20 centipoise to about 1,000 centipoise. In an embodiment, the concentrated hair care composition described herein may have a liquid phase viscosity of from about 200 centipoise to about 15,000 centipoise, alternatively from about 300 centipoise to about 12,000 centipoise, alternatively from about 400 centipoise to about 8,000 centipoise, alternatively from about 500 centipoise to about 5,000 centipoise, and alternatively from about 600 centipoise to about 2,500 centipoise, and alternatively from about 700 centipoise to about 2,000 centipoise.

The viscosity values may be measured employing any suitable rheometer or viscometer at 25.0 °C and at a shear rate of about 2 reciprocal seconds. The viscosities reported herein were measured a Cone/Plate Controlled Stress Brookfield Rheometer R/S Plus, by Brookfield Engineering Laboratories, Stoughton, MA. The cone used (Spindle C-75-1) has a diameter of 75 mm and 1° angle. The viscosity is determined using a steady state flow experiment at constant shear rate of 2 s<sup>-1</sup> and at temperature of 25.0 °C. The sample size is 2.5ml and the total measurement reading time is 3 minutes. The liquid phase viscosity may be measured under ambient conditions and prior to the addition of the propellant.

## J. Optional Ingredients

The concentrated hair care composition described herein may comprise one or more additional components known for use in hair care or personal care products, provided that the additional components are physically and chemically compatible with the essential components described herein, or do not otherwise unduly impair product stability, aesthetics or performance. Such optional ingredients are most typically those materials approved for use in cosmetics and that are described in reference books such as the CTFA Cosmetic Ingredient Handbook, Second Edition, The Cosmetic, Toiletries, and Fragrance Association, Inc. 1988, 1992. Individual concentrations of such additional components may range from about 0.001 wt% to about 10 wt%, by weight of the concentrated hair care composition.

Emulsifiers which may be suitable as an optional ingredient herein include mono- and diglycerides, fatty alcohols, polyglycerol esters, propylene glycol esters, sorbitan esters and other emulsifiers known or otherwise commonly used to stabilize air interfaces, as for example those used during preparation of aerated foodstuffs such as cakes and other baked goods and confectionary products, or the stabilization of cosmetics such as hair mousses.

Further non-limiting examples of such optional ingredients include preservatives, perfumes or fragrances, cationic polymers, viscosity modifiers, coloring agents or dyes, conditioning agents, hair bleaching agents, thickeners, moisturizers, foam boosters, additional surfactants or nonionic cosurfactants, emollients, pharmaceutical actives, vitamins or nutrients, sunscreens, deodorants, sensates, plant extracts, nutrients, astringents, cosmetic particles, absorbent particles, adhesive particles, hair fixatives, fibers, reactive agents, skin lightening agents, skin tanning agents, anti-dandruff agents, perfumes, exfoliating agents, acids, bases, humectants, enzymes, suspending agents, pH modifiers, hair colorants, hair perming agents, pigment particles, anti-acne agents, anti-microbial agents, sunscreens, tanning agents, exfoliation particles, hair growth or restorer agents, insect repellents, shaving lotion agents, non-volatile solvents or diluents (water-soluble and water-insoluble), co-solvents or other additional solvents, and similar other materials.

## K. Foam Dispenser

The foam dispenser may be a mechanical or an aerosol foam dispenser. The foam dispenser comprises a reservoir for holding the concentrated hair care composition. The reservoir may be made out of any suitable material selected from the group consisting of plastic, metal, alloy, laminate, and combinations thereof. In an embodiment, the reservoir may be for one-time



use. In an embodiment, the reservoir may be removable from the foam dispenser. Alternatively, the reservoir may be integrated with the foam dispenser. In an embodiment, there may be two or more reservoirs.

In an embodiment, the reservoir may be comprised of a material selected from the group consisting of rigid materials, flexible materials, and combinations thereof. The reservoir may be comprised of a rigid material if it does not collapse under external atmospheric pressure when it is subject to an interior partial vacuum.

In an embodiment, the foam dispenser may comprise a dip-tube to enable upright dispensing.

In an embodiment, the foam dispenser may be an aerosol foam dispenser of the bag on valve type wherein the container comprises an inner bag and an outer container, which encloses the inner bag, while the inner bag has a valve mechanism attached which is movable between an open position and a closed position. The outer container may be formed from metal or plastic or the like, and any of the propellants described herein can be filled in a space between the outer container and the inner bag. The inner bag may be flexible, and can be made from a single material or from a composite material including plastic, which may comprise at least a polymeric layer and a layer which acts as a gas barrier, e.g., made from metal, such as Aluminum. The inner material of the bag may be inert to the contents of the composition, and the inner material may also be impenetrable by the contents of the composition in the bag. The inner bag may comprise a layer of a material which is essentially impermeable to the propellant inside of the bag. The inner bag may comprise a layer of a material which is essentially impermeable to the propellant outside of the bag which generally is not intended to be mixed with the composition in the inner bag during storage.

In an embodiment, the foam has a dosage weight of from about 1 g to about 5 g when dispensed from the foam dispenser. In another embodiment, the foam has a dosage weight of from about 1 g to about 7 g when dispensed from the foam dispenser, alternatively from about 2 g to about 6 g, alternatively from about 2.5 g to about 5 g, and alternatively from about 3 g to about 4.5 g. The dosage may be obtained via a single squeeze or actuation or pump of the foam dispenser, but may also be accomplished via two squeezes or actuations or pumps of the foam dispenser.

### L. Propellant

The concentrated hair care composition described herein may comprise from about from about 1% to about 6% propellant, alternatively from about 2% to about 5% propellant, and alternatively from about 3% to about 4% propellant, by weight of the concentrated hair care composition. In an embodiment, the concentrated hair care composition described herein may comprise from about from about 1% to about 12% propellant, alternatively from about 2% to about 10% propellant, alternatively from about 3% to about 8% propellant, and alternatively from about 4% to about 6% propellant, by weight of the concentrated hair care composition.

The propellant may comprise one or more volatile materials, which in a gaseous state, may carry the other components of the concentrated hair care composition in particulate or droplet form. The propellant may have a boiling point within the range of from about  $-45^{\circ}\text{C}$ . to about  $5^{\circ}\text{C}$ . The propellant may be liquefied when packaged in convention aerosol containers under pressure. The rapid boiling of the propellant upon leaving the aerosol foam dispenser may aid in the atomization of the other components of the concentrated hair care composition.

Aerosol propellants which may be employed in the aerosol composition may include the chemically-inert hydrocarbons such as propane, n-butane, isobutane, cyclopropane, and mixtures thereof, as well as halogenated hydrocarbons such as dichlorodifluoromethane, 1,1-dichloro-1,1,2,2-tetrafluoroethane, 1-chloro-1,1-difluoro-2,2-trifluoroethane, 1-chloro-1,1-difluoroethylene, 1,1-difluoroethane, dimethyl ether, monochlorodifluoromethane, trans-1,3,3,3-tetrafluoropropene, and mixtures thereof. The propellant may comprise hydrocarbons such as isobutane, propane, and butane—these materials may be used for their low ozone reactivity and may be used as individual components where their vapor pressures at  $21.1^{\circ}\text{C}$ . range from about 1.17 Bar to about 7.45 Bar, alternatively from about 1.17 Bar to about 4.83 Bar, and alternatively from about 2.14 Bar to about 3.79 Bar.

### M. Foam Density

The concentrated hair care composition may be dispensed as a foam wherein the foam has a density of from about  $0.025\text{ g/cm}^3$  to about  $0.30\text{ g/cm}^3$ , alternatively from about  $0.035\text{ g/cm}^3$  to about  $0.20\text{ g/cm}^3$ , alternatively from about  $0.045\text{ g/cm}^3$  to about  $0.15\text{ g/cm}^3$ , and alternatively from about  $0.055\text{ g/cm}^3$  to about  $0.12\text{ g/cm}^3$ . In an embodiment, the concentrated hair care composition may be dispensed as a foam wherein the foam as a density of from about  $0.025\text{ g/cm}^3$  to about  $0.40\text{ g/cm}^3$ , alternatively from about  $0.035\text{ g/cm}^3$  to about  $0.30\text{ g/cm}^3$ , alternatively

from about  $0.045 \text{ g/cm}^3$  to about  $0.20 \text{ g/cm}^3$ , and alternatively from about  $0.055 \text{ g/cm}^3$  to about  $0.15 \text{ g/cm}^3$ .

#### **N. Carrier**

5 The concentrated hair care composition described herein may comprise from about from about 60% to about 90%, alternatively from about 65% to about 87.5%, alternatively from about 67.5% to about 85%, alternatively from about 70% to about 82.5%, and alternatively from about 72.5% to about 80% of a carrier, by weight of the concentrated hair care composition. In an embodiment, the carrier is water.

#### **O. Method of Treating Hair**

10 The method of treating the hair described herein comprises (1) providing a concentrated hair care composition, as described herein, in a foam dispenser; (2) dispensing the concentrated hair care composition from the foam dispenser as a dosage of foam; (3) applying the foam to the  
15 hair, wherein the one or more silicones are deposited onto the hair when applying the foam to the hair; and (4) rinsing the foam from the hair; wherein the one or more silicones forms a silicone layer on the hair. In an embodiment, the concentrated hair care composition is a rinse-off hair care composition.

#### **P. Silicone Layer**

20 In an embodiment, the silicone layer may have a Hair Damage Repair Index of from about 0.8 to about 4.0, alternatively from about 1.0 to about 3.8, alternatively from about 1.2 to about 3.6, alternatively from about 1.4 to about 3.4, alternatively from about 1.6 to about 3.2, alternatively from about 1.8 to about 3.0, and alternatively from about 2.0 to about 2.8.

25 In an embodiment, the hair has a water advancing contact angle, and after the method of treating hair described herein, the water advancing contact angle is increased to a value of about 104 degrees to about 125 degrees, alternatively about 106 degrees to about 124 degrees, alternatively about 108 degrees to about 123 degrees, alternatively about 110 degrees to about 122 degrees, alternatively about 112 degrees to about 121 degrees, and alternatively about 114  
30 degrees to about 120 degrees.

In an embodiment, the silicone layer may have a normalized thickness of deposition (nm/ppm) of from about 0.001 to about 0.019, alternatively from about 0.003 to about 0.016, alternatively from about 0.006 to about 0.013, and alternatively from about 0.008 to about 0.011.

In an embodiment, the silicone layer may have a normalized coefficient of variation of deposition thickness (%/ppm) of from about 0.001 to about 0.075, alternatively from about 0.003 to about 0.070, alternatively from about 0.006 to about 0.065, alternatively from about 0.009 to about 0.060, and alternatively from about 0.012 to about 0.055.

In an embodiment, the silicone layer may have a normalized maximum thickness of deposition (nm/ppm) of from about 0.005 to about 0.25, alternatively from about 0.01 to about 0.20, alternatively from about 0.015 to about 0.15, alternatively from about 0.02 to about 0.10, alternatively from about 0.025 to about 0.09, and alternatively from about 0.03 to about 0.08.

In an embodiment, the silicone layer has a Targeted Deposition Index of from about 0.8 to about 1.6, alternatively from about 0.9 to about 1.5, alternatively from about 1.0 to about 1.4, alternatively from about 1.05 to about 1.35, alternatively from about 1.10 to about 1.30, alternatively from about 0.85 to about 1.30, alternatively from about 0.90 to about 1.30, alternatively from about 0.95 to about 1.30, alternatively from about 1.0 to about 1.30, alternatively from about 1.10 to about 1.30, and alternatively from about 1.15 to about 1.30.

In an embodiment, the silicone layer has a % Targeted Deposition of from about -20% to about 50%, alternatively from about -10% to about 40%, alternatively from about 0% to about 35%, alternatively from about 5% to about 35%, alternatively from about 10% to about 25%, alternatively from about -20% to about 30%, alternatively from about -10% to about 30%, alternatively from about 0% to about 30%, alternatively from about 5% to about 30%, alternatively from about 10% to about 30%, and alternatively from about 15% to about 30%.

#### EXAMPLES & DATA

The following examples illustrate the concentrated hair care composition described herein. The exemplified compositions can be prepared by conventional formulation and mixing techniques. It will be appreciated that other modifications of the present invention within the skill of those in the shampoo formulation art can be undertaken without departing from the spirit and scope of this invention. All parts, percentages, and ratios herein are by weight unless otherwise specified. Some components may come from suppliers as dilute solutions. The amount stated reflects the weight percent of the active material, unless otherwise specified.

Three "Clarifying" shampoos are employed in the below examples that were void of high melting point fatty compounds and conditioning agents. One was a Pantene clarifying shampoo and the other two were concentrated foam shampoos. The concentrated foam shampoos may be prepared by mixing together water and surfactants along with any solids that need to be melted at

an elevated temperature, e.g. about 75°C. The ingredients are mixed thoroughly at the elevated temperature and then cooled to ambient temperature. Additional ingredients, including electrolytes, polymers, silicone emulsions, preservatives and fragrances may be added to the cooled product.

Table 1: Concentrated Aerosol Foam Cleansing Shampoo Composition

Raw Material	Foam Shampoo 1	Foam Shampoo 2
Sodium Undecyl Sulfate (C11 70% active) <sup>1</sup>	23.6	
Lauramidopropyl Betaine (LAPB 35% active) <sup>2</sup>	5.8	
Para Hydroxy Phenyl Butanone <sup>3</sup>	3.9	
Alkyl polyglucoside <sup>4</sup>		25.2
Cocamidopropyl betaine		3.9
Polyvinyl alcohol <sup>5</sup>	1.9	1.9
Perfume	2.3	2.3
Citric Acid	0.3	0.3
Preservative (Kathon)	0.03	0.03
Propellant (Aeron-46)	3.1	3.1
Water	(q.s.)	(q.s.)
Weight % of high melting point fatty compounds	0%	0%

1. Sodium Undecyl Sulfate (C11, Isachem 123S) at 70% active, supplier: P&G
2. LAPB (Mackam DAB), at 35% active level, supplier: Rhodia
3. Raspberry Ketone, supplier: Spectrum
4. EcoSense 919 available from Dow Chemical.
5. PVA-403 available from Kuraray

The following aerosol conditioner compositions may be prepared by weighing distilled water and the aminosilicone emulsions into a stainless steel beaker. The beaker is placed in a water bath on a hot plate while mixing with overhead mixer at 100 to 150 rpm. If fatty alcohols are present in the formula, the cetyl alcohol and stearyl alcohol are added and the mixture is heated to 70-75°C. Cetyltrimethylammonium chloride is then added and mixing speed is increased to 250-350 rpm due to viscosity increase. When the materials are all heated thoroughly and homogenous, the heating is stopped while the mixture is continued to stir. The batch is cooled to 35°C by removing the hot water from the water bath and replacing with cold water. The perfume

and Kathon are added and with continued stirring for ~ 10 minutes. For foaming, propellant Aeron-46 was added to each of the below formulas at a weight ratio of 4 parts Aeron-46 to 96 parts of formula within an aerosol container.

The ability to foam was assessed by shaking the aerosol container for 10 seconds and then seeing if 5 grams could be dispensed into a weigh boat. The foam quality was assessed by spreading the foam and assessing the ability to spread without foam collapse on a qualitative scale (+++ excellent, ++ good, + fair, - poor).

**Table 2: Concentrated Aerosol Foam Conditioner Composition**

Raw Material	Ex 1	Ex 2	Ex 3	Ex 4	Ex 5	Ex 6	Ex 7
Aminosilicone <sup>1</sup>	12	12	12	12	12	12	12
Perfume	2.4	2.4	3.0	3.0	3.0	3.0	3.0
Cetyltrimethylammonium Chloride		2.5	2.5	2.5	2.5	2.5	2.5
Cetyl Alcohol			1.5	3.0	4.0	6.0	9
Stearyl Alcohol			1.5	3.0	4.0	6.0	9
Preservative (Kathon)	0.03	0.03	0.03	0.03	0.03	0.03	0.03
Water	(q.s.)	(q.s.)	(q.s.)	(q.s.)	(q.s.)	(q.s.)	(q.s.)
Weight ratio of oil to high melting point fatty compounds	100:0	100:0	80:20	67:33	60:40	50:50	40:60
Viscosity (cp)	< 200	< 200	810	1740	5,450	12,900	33,400
Ability to dispense foam	Yes	Yes	Yes	Yes	Yes	Yes	No
Foam Quality	+++	+++	+++	+++	+	+	None

1 Silsoft 253 (20% active) nano-emulsion available from Momentive (10-20nm)

The foam conditioner compositions may be concentrated in silicone (12% by weight) as it has been determined that consumers dose low density foams at approximately 1/3 the dosage of normal conditioners. Accordingly, 12% silicone delivered from the foam delivers about the same dosage of silicone as a normal 4% silicone liquid conditioner. As demonstrated in Table 2, at such a concentrated level of silicone, as the ratio of oil to high melting point fatty compounds decreases, the ability to dispense foam and deliver good foam quality decreases. This is also correlating to viscosity. Importantly, below an oil to high melting point fatty compound ratio of 50:50, the foam was not able to be dispensed (formula was too viscous). Additionally, the ratios of 50:50 and 60:40 had reduced foam quality versus the higher ratios. Accordingly, Examples 1

through 4, with both excellent dispensing and foam quality, were progressed for performance testing as described below.

Concentrated foam conditioners 1-4 in Table 2 were treated onto General Population brown hair switches and dyed hair (dyed with a commercially available level 3 oxidative dye) as part of a regimen with Pantene Pro-V Clarifying Shampoo for up to 6 treatment cycles. Example 2 foam conditioner was also paired with the foam shampoo 1 and foam shampoo 2. As a regimen control, the Pantene Pro-V Clarifying Shampoo was combined with Pantene Anti-Breakage Conditioner. The latter is known to have an aminosilicone content of 2.5% and a total high melting point fatty compounds (cetyl and stearyl alcohols) content of 5.20% for a weight ratio of oil to high melting point fatty compounds of 32.5:67.5. Deposition data, wet and dry combing data, and hair volume data was collected on the hair switches after 6 treatment cycles. Scanning electron microscopy, hair/water contact angles and Atomic Force Microscopy measurements were taken on the hair switches after 6 cycles.

Multiple Cycle Shampoo plus Conditioner treatments:

1. Six 4 gram, 8 inch General Population brown hair switches are wet with 100 degrees Fahrenheit water at a sink (bound on root-ends with glue/tape and hanging on metal holder) with a shower head fixture (flow rate is 1.5 gallons per minute) for 15 to 20 seconds.
2. Liquid shampoos are applied at 0.1grams of product per gram of hair (e.g., Pantene Pro-V Clarifying Shampoo) via a syringe and milked/scrubbed for 30 seconds followed by a 30 seconds shower head rinse (with gentle manipulation at top of switch to ensure uniform rinsing). Concentrated liquid foam shampoos are applied at 0.05 grams of product per gram of hair with a spatula (foam is dispensed in weigh boat and applied weight recorded) and following the same application procedure.
3. Liquid conditioners are applied at a 0.1 grams of product per gram of hair (e.g., Pantene Moisture Renewal Conditioner etc.) via a syringe (weighed on weigh scale) evenly over the hair switch and milked/scrubbed for 30 seconds followed by a 30 seconds shower rinse (with gentle manipulation at top of switch to ensure uniform rinsing). Concentrated liquid foam conditioners are applied at 0.033 grams of product per gram of hair with a spatula (foam is dispensed in weigh boat and applied weight recorded) and following the same application procedure.

4. The hair is then dried in a heat box set at 60C for ~45 minutes or until mostly dry before starting the next treatment cycle or the completion of the treatment cycles.

For multiple cycle testing, the above procedure is repeated for a set number of times. For instance, for a six cycle test, the above steps 1-4 are repeated six times.

#### Deposition Data and Deposition Purity (6 treatment cycles):

Deposition Purity may be determined by the ratio of silicone deposited per weight of hair to the total deposition of other ingredients per weight of hair. Silicone may be determined by digestion of the hair followed by an analysis with a quantitative elemental technique such as ICP for total silicon and converting to silicone based on the % of silicon in the silicone by weight. The total deposition may be determined by the sum of separate deposition measurements. The separate deposition measurements may include but are not limited to: fatty alcohols, EGDS, quaternized agents and silicone. Typically these measurements involve extracting the hair then separating the ingredients of interest with chromatography and quantifying with an externally calibration based on test solution concentration.

#### ICP-OES silicone hair digestion method:

Hair samples treated with different products are submitted as balls of hair with an average sample size of 0.1 g. These hair samples are then digested using a single reaction chamber microwave digestion system (Milestone Inc., Shelton, CT) using a 6:1 HNO<sub>3</sub>:H<sub>2</sub>O<sub>2</sub> mixture and an aliquot of methyl isobutyl ketone (MIBK) in Teflon digestion vessels. A gentle digestion program with a ramp to 95°C and a manual vent after cooling below 30°C is used to facilitate retention of silicon. After dilution to volume, the samples are run against an inorganic silicon calibration curve produced on an Optima 8300 ICP-OES system (Perkin Elmer, Waltham, Massachusetts) run in the axial mode. The silicon values determined are converted to a concentration of silicone polymer-equivalents deposited on the hair sample using the theoretical silicon concentration of the polymer provided by the manufacturer. An untreated hair sample is analyzed to determine the background concentration of silicon to allow correction if needed. Another untreated hair sample is spiked with a known amount of polymer and analyzed to ensure recovery of the polymer and verify the analysis.



General Population HairTable 3: Results of Deposition and Deposition Purity on General Population Hair after 6 treatment cycles

Regimen	Fatty Alcohol Deposition (ppm)	Amino-silicone Deposition (ppm) [%RSD]	Alkyl Quat Deposition (ppm)	Amino-silicone-to-fatty alcohol ratio	Oil Deposition Purity	Total Deposition (ppm)
Clarifying Shampoo plus Pantene Anti-breakage Conditioner	1749 +/- 81	1557 +/- 844 [54%]	26 +/- 2	0.9	47%	3332
Clarifying Shampoo plus Ex 1 Conditioner	3.0 +/- 8	1341 +/- 449 [33%]	6 +/- 0.4	447	99%	1350
Clarifying Shampoo plus Ex 2 Conditioner	14 +/- 4	1040 +/- 105 [10%]	140 +/- 13	74	87%	1194
Clarifying Shampoo plus Ex 3 Conditioner	488 +/- 23	1334 +/- 82 [6%]	94 +/- 13	2.7	70%	1916
Clarifying Shampoo plus Ex 4 Conditioner	668 +/- 36	1546 +/- 67 [4%]	67 +/- 10	2.3	68%	2281
Foam Shampoo 1 plus Ex 2 Conditioner	8 +/- 3	934 +/- 60 [6%]	126 +/- 9	117	87%	1068
Foam Shampoo 2 plus Ex 2 Conditioner	1 +/- 5	996 +/- 58 [4%]	158 +/- 12	996	86%	1155

The Table 3 deposition data on general population hair after 6 treatment cycles demonstrates the regimens involving a foam conditioner of the present invention deposit high levels of aminosilicone onto hair (900 to 1,600ppm versus 1,600ppm for the liquids control regimen), but importantly with significantly less fatty alcohol co-deposits (1 to 700ppm fatty alcohols versus 1,557ppm for the liquid control regimen). Correspondingly, the measured oil deposition purity was much higher for the regimens of the present invention (68% to 99% purity) versus the liquid regimen control (47% purity).

Dyed HairTable 4: Results of Deposition and Deposition Purity on Dyed Hair after 6 treatment cycles

Regimen	Fatty Alcohol Deposition (ppm)	Amino-silicone Deposition (ppm)	Alkyl Quat Deposition (ppm)	Amino-silicone-to-fatty alcohol ratio	Oil Deposition Purity	Total Deposition (ppm)	Dyed-to-General Population Hair Deposition Ratio (x 100 %)
Clarifying Shampoo plus Pantene Anti-breakage Conditioner	1532 +/- 84	1057 +/- 260 [25%]	32 +/- 3	0.7	40%	2621	68%
Clarifying Shampoo plus Ex 1 Conditioner	31 +/- 16	1464 +/- 78 [5%]	11 +/- 0	47	97%	1506	109%

Clarifying Shampoo plus Ex 2 Conditioner	31 +/- 16	1385 +/- 149 [11%]	149 +/- 26	45	88%	1565	133%
Clarifying Shampoo plus Ex 3 Conditioner	444 +/- 12	2040 +/- 147 [7%]	115 +/- 25	4.6	78%	2599	153%
Clarifying Shampoo plus Ex 4 Conditioner	636 +/- 41	2225 +/- 108 [5%]	84 +/- 13	3.5	76%	2945	144%
Foam Shampoo 1 plus Ex 2 Conditioner	7 +/- 17	1484 +/- 47 [3%]	133 +/- 15	212	91%	1624	159%
Foam Shampoo 2 plus Ex 2 Conditioner	9 +/- 10	1479 +/- 103 [7%]	195 +/- 16	164	88%	1683	148%

The Table 4 deposition data on dyed hair after 6 treatment cycles demonstrates the regimens involving a foam conditioner of the present invention to deposit high levels of aminosilicone onto hair (1,385 to 2,225ppm versus 1,057ppm for the liquids control regimen), but importantly with significantly less fatty alcohol co-deposits (7 to 636ppm fatty alcohols versus 1,557ppm for the liquid control regimen). Correspondingly, the measured oil deposition purity was much higher for the regimens of the present invention (76% to 97% purity) versus the liquid regimen control (40% purity). Moreover, the regimens involving a foam conditioner of the present invention also deposit significantly greater amount of silicone onto the more polar dyed hair than on general population hair (dyed to general population hair deposition ratios of 109% to 159%) versus the liquid regimen control which deposited less silicone on the more polar dyed hair (dyed to general population hair deposition ratios of 68%).

General Population Hair Wet Combing, Dry Combing and Hair Volume Data (6 treatment cycles):

Wet combing, dry combing and hair volume was assessed of the hair tresses after the 6 treatment cycles via a sensory panel encompassing 12 individuals.

Wet Combing Test (on the day of the final treatment cycle):

After the last treatment cycle, the treated hair tresses were wrapped in aluminum foil and labeled in groups. During the panel, a hair tress from each leg grouping was hung on a metal bar and with each switch being detangled with the wider spacing teeth on a professional comb. The panelists then evaluated the ease of wet combing of the switches using the 'small end' of a professional comb (using gloved hand to stabilize switch while combing if needed) and record scores on the provided evaluation form (0-10 scale). After all 5 sets of hair have been combed (2 panelists per hair set), hang carts with hair in CT room (50% RH, 70F).

Dry Combing Test (at least one day after the wet combing test):

The dried hair switches from each treatment group were placed in separate metal holders hanging side by side on a metal bar. The panelists evaluated the ease of dry combing of the switches using the 'small end' of a professional comb and record scores on the provided evaluation form (0-10 scale; 2 panelists per hair set).

General Population HairTable 5: Wet/Dry Combing and Hair Volume on General Population Hair after 6 treatment cycles

Regimen	Regimen weight ratio of oil to high melting point fatty compounds	Wet Combing	Dry Combing	Hair Volume
Clarifying Shampoo Control	--	2.0	2.6	8.5
Clarifying Shampoo plus Pantene Anti-breakage Conditioner	32.5:67.5	8.2	9.8	4.3
Clarifying Shampoo plus Ex 1 Conditioner	100:0	9.0	8.0	7.6
Clarifying Shampoo plus Ex 2 Conditioner	100:0	9.4	8.2	5.6
Clarifying Shampoo plus Ex 3 Conditioner	80:20	9.3	8.6	4.6
Clarifying Shampoo plus Ex 4 Conditioner	67:33	9.2	8.3	3.6
Foam Shampoo 1 plus Ex 2 Conditioner	100:0	8.6	7.1	6.0
Foam Shampoo 2 plus Ex 2 Conditioner	100:0	8.9	7.5	7.5

The above data on general population hair after 6 treatment cycles demonstrates the regimens involving a foam conditioner of the present invention provide acceptable wet combing performance (from 8.2 to 9.4 average scores) and dry combing performance (from 7.1 to 8.6 average scores) versus the liquid control regimen (wet combing of 8.2 and dry combing of 9.8). But, importantly the regimens involving a foam conditioner of the present invention were able to do this with very good hair volume performance after the end of the treatment cycles (hair volume average scores of 3.6 to 7.6) relative to the liquid regimen control (hair volume of 4.3). Also, the hair volume trends with the weight ratio of oil to high melting point fatty compounds within the regimen compositions (with 100:0 ratios providing the best hair volume performance). This is hypothesized to be due to significantly less co-deposits of high melting point fatty compounds.

Dyed HairTable 6: Wet/Dry Combing and Hair Volume on Dyed Hair after 6 treatment cycles

Regimen	Regimen weight ratio of oil to high melting point fatty compounds	Wet Combing	Dry Combing	Hair Volume
Clarifying Shampoo Control	--	1.6	1.5	5.5
Clarifying Shampoo plus Pantene Anti-breakage Conditioner	32.5:67.5	8.0	9.6	4.1
Clarifying Shampoo plus Ex 1 Conditioner	100:0	9.3	7.6	8.1
Clarifying Shampoo plus Ex 2 Conditioner	100:0	9.8	8.5	4.3
Clarifying Shampoo plus Ex 3 Conditioner	80:20	9.4	8.5	5.9
Clarifying Shampoo plus Ex 4 Conditioner	67:33	9.3	8.7	3.9
Foam Shampoo 1 plus Ex 2 Conditioner	100:0	8.7	7.7	6.2
Foam Shampoo 2 plus Ex 2 Conditioner	100:0	8.6	7.5	7.1

\*representative of oxidatively damaged hair

The Table 6 data on dyed hair after 6 treatment cycles demonstrates the regimens involving a foam conditioner of the present invention to provide very good wet combing performance (from 8.6 to 9.3 average scores) and dry combing performance (from 7.5 to 8.5 average scores) comparable to the liquid control regimen (wet combing of 8.0 and dry combing of 9.6). But, importantly the regimens involving a foam conditioner of the present invention were able to do this with good hair volume performance after the end of the treatment cycles (hair volume average scores of 3.9 to 8.1) relative to the liquid regimen control (hair volume of 4.1). Also, the hair volume trends with the weight ratio of oil to high melting point fatty compounds within the regimen compositions (with 100:0 ratios providing the best hair volume performance). This is hypothesized to be due to significantly less co-deposits of high melting point fatty compounds.

Scanning Electron Microscopy (6 treatment cycles)

Ten to twelve general population hair strands with 1cm length hair from each treatment were mounted on SEM sample holder, coated with Au/Pd for 45 seconds for conductivity, transferred sample holder into SEM chamber, and used Hitachi S4700 Field Emission High Resolution SEM for imaging analysis at 3kv with built-in Bruker Quantax Esprit SDD for EDS (Energy Dispersive X-ray Spectrometry) analysis for elemental information at 5kv. The high-resolution image visualized the details of topography, hair structure and the deposition on its surface. EDS revealed the existence of elements of and correlated to the image topography.

The SEM images after 6 treatment cycles on general population hair in Figures 1-7 demonstrate the regimens involving a foam conditioner as described herein to provide surface

deposits with improved morphology (thinner, smoother and more even deposition) versus the liquid control regimen (irregular deposits that are thicker, not smooth and lacking even deposition). This may be due to significantly less co-deposits of high melting point fatty compounds (from 68% to 99% silicone deposition purity) versus the liquid regimen control (from 40% to 47% silicone deposition purity).

#### Additional Examples:

The following aerosol conditioner compositions in Tables 7 and 8 may be prepared by weighing distilled water and the aminosilicone emulsions into a stainless steel beaker. The beaker is placed in a water bath on a hot plate while mixing with overhead mixer at 100 to 150 rpm. If fatty alcohols are present in the formula, the cetyl alcohol and stearyl alcohol are added and the mixture is heated to 70-75C. The behentrimonium methosulfate is then added and mixing speed is increased to 250-350 rpm due to viscosity increase. When the materials are all heated thoroughly and homogenous, the heating is stopped while the mixture is continued to stir. The batch is cooled to 35C by removing the hot water from the water bath and replacing with cold water. The perfume and Kathon are added and with continued stirring for ~ 10 minutes. For foaming, propellant Aeron-46 is added to each of the below formulas at a weight ratio of 4 parts Aeron-46 to 96 parts of formula within an aerosol container.

Table 7

Raw Material	Ex 8	Ex 9	Ex 10	Ex 11	Ex 12	Ex 13
Aminosilicone <sup>1</sup>	8	4	2	0	8	4
Aminosilicone <sup>2</sup>		4	2	4		4
Perfume	2.0	2.0	2.0	2.0	2.0	2.0
Behentrimonium methosulfate	4.3	4.3	4.3	4.3	4.3	4.3
Cetyl Alcohol	0.86	0.86	0.86	0.86	0.86	0.86
Stearyl Alcohol	2.15	2.15	2.15	2.15	2.15	2.15
Hydroxyethyl cellulose <sup>3</sup>	0.00	0.00	0.00	0.00	1.00	0.5
Citric Acid	0.02	0.02	0.02	0.02	0.02	0.02
Benzyl Alcohol	0.4	0.4	0.4	0.4	0.4	0.4
Disodium EDTA	0.13	0.13	0.13	0.13	0.13	0.13
Preservative (Kathon)	0.03	0.03	0.03	0.03	0.03	0.03
Water	(q.s.)	(q.s.)	(q.s.)	(q.s.)	(q.s.)	(q.s.)

Weight ratio of oil to high melting point fatty compounds	73:27	73:27	57:43	57:43	73:27	73:27
Viscosity (cp)	591	756	1461	9505	6830	3202

- 1 Silsoft 253 (20% active) nano-emulsion available from Momentive (10-20nm)
- 2 Y17045 (100% active) available experimentally from Momentive
- 3 Natrosol 250HHR available from Ashland Chemicals.

Table 8

Raw Material	Ex 14	Ex 15	Ex 16	Ex 17	Ex 18	Ex 19
Amino morpholino silicone <sup>4</sup>	8	12	16			
Aminosilicone <sup>5</sup>				8	12	16
Perfume	2.0	2.0	2.0	2.0	2.0	2.0
Behentrimonium methosulfate	4.3	4.3	4.3	4.3	4.3	4.3
Cetyl Alcohol	0.86	0.86	0.86	0.86	0.86	0.86
Stearyl Alcohol	2.15	2.15	2.15	2.15	2.15	2.15
Citric Acid	0.02	0.02	0.02	0.02	0.02	0.02
Benzyl Alcohol	0.4	0.4	0.4	0.4	0.4	0.4
Disodium EDTA	0.13	0.13	0.13	0.13	0.13	0.13
Preservative (Kathon)	0.03	0.03	0.03	0.03	0.03	0.03
Water	(q.s.)	(q.s.)	(q.s.)	(q.s.)	(q.s.)	(q.s.)
Weight ratio of oil to high melting point fatty compounds	73:27	80:20	84:16	73:27	80:20	84:16

4. BELSIL® ADM 8301 E (20% active) nano-emulsion available from Wacker (< 50 nm)
5. CE-8170 Microemulsion (20% active) available from Dow Corning (< 50 nm)

### **Advancing Contact Angle Hair Damaged Repair Index**

5

#### **Concentrated Hair Shampoo Foam Example 1**

The shampoo composition in Table 9 was made by mixing together water, dipropylene glycol, the cationic polymer, and surfactants along with any solids that need to be melted at an elevated temperature, e.g. about 75°C. The ingredients are mixed thoroughly at the elevated temperature and then cooled to ambient temperature. The perfume and Kathon are added with continued stirring for ~ 10 minutes. For foaming, the 94 parts of the batch is transferred to appropriate container and 6 parts of hydrofluoroolefin (HFO-1234ze from Honeywell) is added.

Table 9

Raw Material	Ex 1
Sodium Undecyl Sulfate <sup>1</sup>	24.0
Lauramidopropyl Betaine <sup>2</sup>	6.0
Guar, Hydroxypropyl Trimonium Chloride <sup>3</sup>	0.4
Dipropylene Glycol	4.0
Perfume	2.4
Preservative (Kathon)	0.03
Water (q.s.)	

- 5      1. Sodium Undecyl Sulfate (C11, Isachem 123S) at 70% active, supplier: P&G  
        2. LAPB (Mackam DAB), at 35% active level, supplier: Rhodia  
        3. Jaguar C500, MW of 500,000, CD of 0.8, from Rhodia  
        4. Hydrofluoroolefins (HFO-1234ze) from Honeywell

10

**Concentrated Hair Conditioner Foam Examples 2, 3 & 4**

The inventive conditioner compositions in Table 10 were prepared by weighing distilled water and the oil (aminosilicone) emulsions into a stainless steel beaker. The beaker is placed in a water bath on a hot plate while mixing with overhead mixer at 100 to 150 rpm. If fatty alcohols are present in the formula, the cetyl alcohol and stearyl alcohol are added and the mixture is heated to 70-75C. Cetyltrimethylammonium chloride is then added and mixing speed is increased to 250-350 rpm due to viscosity increase. When the materials are all heated thoroughly and homogenous, the heating is stopped while the mixture is continued to stir. The batch is cooled to 35C by removing the hot water from the water bath and replacing with cold water. The perfume and Kathon are added and with continued stirring for ~ 10 minutes. For foaming, the 94 parts of the batch is transferred to appropriate container and 6 parts of propellant Aeron-46 (Isobutane/Propane at 84.8/15.2 available from Diversified CPC International) is added.

Table 10

Raw Material	Ex 2	Ex 3	Ex 4
Aminopropylaminoethylpolysiloxane <sup>1</sup>	16	12	12
Perfume	2.4	2.4	2.4
Cetrimonium chloride <sup>2</sup>	2.5	1.0	
Behentrimonium chloride <sup>3</sup>		1.5	

Distearyldimonium chloride <sup>4</sup>		1.0	
Cetyl Alcohol	1.35		
Stearyl Alcohol	3.15		
Preservative (Kathon)	0.03	0.03	0.03
Water (q.s.)			
Weight ratio of oil to high melting point fatty compounds	78:22	100:0	100:0

- 1 Aminosilicone micro-emulsion (Silsoft 253), available from Momentive
- 2 CTAC (Varisoft 100), available from Evonik
- 3 BTMAC (Genamin KDMP), available from Clariant
- 4 DSDMC (Varisoft TA100), available from Evonik
- 5 A46 (Isobutane/Propane = 84.8/15.2) available from Diversified CPC International

A 9 leg multi-cycle hair treatment study was conducted including the Table 9 foam shampoo Example 1 and Table 10 conditioner Examples 2 through 4 and a selection of currently marketed shampoo and conditioning products (the shampoo and conditioner product pairings as shown in Table 11). Due to the difference in product densities, the foam products were treated at a lower dosage (0.05 and 0.033 grams of product per gram of hair) versus the liquid products (0.1 grams of product per gram of hair). The study was conducted through 20 treatment cycles with data being collected at cycles 1, 5, 10 and 20.

10 Table 11

<b>Leg</b>	<b>Shampoo</b>	<b>Shampoo Dosage (g/g hair)</b>	<b>Conditioner</b>	<b>Conditioner Dosage (g/g hair)</b>
1	Pantene <sup>®</sup> Clarifying Shampoo	0.1 g/g	None	--
2	Dove <sup>®</sup> Daily Moisture Shampoo	0.1 g/g	Dove <sup>®</sup> Daily Moisture Conditioner	0.1 g/g
3	Pantene <sup>®</sup> Anti-breakage Shampoo	0.1 g/g	Pantene <sup>®</sup> Anti-Breakage Conditioner	0.1 g/g
4	Pantene <sup>®</sup> Expert Intensive Color Care Shampoo	0.1 g/g	Pantene <sup>®</sup> Repair & Protect Conditioner	0.1 g/g
5	Pantene <sup>®</sup> Expert Intensive Color Care Shampoo	0.1 g/g	Pantene <sup>®</sup> Expert Dry Defy Conditioner	0.1 g/g
6	Example 1 FOAM Shampoo (aerosol)	0.05 g/g	Dove <sup>®</sup> Weightless Moisturizers <sup>™</sup> FOAM Conditioner (aerosol)	0.033 g/g
7	Example 1 FOAM Shampoo (aerosol)	0.05 g/g	Example 2 FOAM Conditioner (aerosol)	0.033 g/g



8	Example 1 FOAM Shampoo (aerosol)	0.05 g/g	Example 3 FOAM Conditioner (aerosol)	0.033 g/g
9	Example 1 FOAM Shampoo (aerosol)	0.05 g/g	Example 4 FOAM Conditioner (aerosol)	0.033 g/g

#### Undamaged Virgin Hair and Chemically Damaged Hair Substrates

The concentrated hair care composition described herein is applicable to hair that has been subject to any degree of consumer meaningful hair damage, i.e. hair damage from known chemical hair treatments (oxidative or reductive or high pH or low pH); hair damage from excessive mechanical wear or abrasion (combing of wet hair, combing/brushing of dry hair, blow dryer bars etc.); hair damage from heat treatments (curling irons, blow dryers, flat irons, curling with heat etc.); hair damage from excessive exposure to UV rays from the sun; hair damage from excessive exposure to chemically treated or sanitized water (chlorine, chlorine dioxide, chloramine, hydrogen peroxide, bromine, etc.); and combinations thereof. The hair damaging chemical hair treatments include, but are not limited to, permanent hair dyes, demi-permanent hair dyes, hair bleaches, hair lightening products, hair permanent wave products, hair relaxer products, or any treatment that subjects hair to extremes in pH (either greater than a pH of 8 with a base such as ammonium hydroxide or less than a pH of 4 with an acid for at least greater than one minute).

In the following data, the hair was chemically damaged with a hair bleaching composition comprising 5.5% hydrogen peroxide, 2% ammonium hydroxide, and 0.2% EDTA. The hair was submerged within this hair bleaching composition for a period of 35 minutes followed by thorough rinsing. The hair was then dried while brushing continuously for 3 minutes (1.5 minutes per side). However, the measured Hair Damage Repair Index is normalized and thereby applicable to any degree of consumer relevant hair damage including subjecting the hair to a bleaching composition for shorter (down to 1 minute) or longer time periods (up to several hours) provided statistically meaningful differences can be measured between the starting undamaged hair and damaged hair substrates.

The hair substrates employed for this study were 4 gram, 8 inch brown virgin (general population) hair tresses available from International Hair Importers & Products (Glendale, NY).

#### Multiple Cycle Shampoo plus Conditioner treatments:

The above shampoo and conditioner pairings from each of the 9 legs were treated onto virgin brown hair and chemically bleached damaged hair through 20 hair treatment cycles:

1. Six 4 gram, 8 inch hair switches (virgin brown hair or chemically bleached damaged hair) are wet with 100 degrees Fahrenheit water at a sink (bound on root-ends with glue/tape and hanging on metal holder) with a shower head fixture (flow rate is 1.5 gallons per minute) for 15 to 20 seconds.
2. Liquid shampoos are applied at 0.1grams of product per gram of hair (e.g., Pantene Pro-V Clarifying Shampoo) via a syringe and milked/scrubbed for 30 seconds followed by a 30 seconds shower head rinse (with gentle manipulation at top of switch to ensure uniform rinsing).
3. Liquid conditioners are applied at 0.1 grams of product per gram of hair (e.g., Pantene Anti-breakage Conditioner etc.) via a syringe (weighed on weigh scale) evenly over the hair switch and milked/scrubbed for 30 seconds followed by a 30 seconds shower rinse (with gentle manipulation at top of switch to ensure uniform rinsing). Concentrated liquid foam conditioners are applied at 0.033 grams of product per gram of hair with a spatula (foam is dispensed in weigh boat and applied weight recorded) and following the same application procedure.
4. The hair is then dried in a heat box set at 60C for ~45 minutes or until mostly dry before starting the next treatment cycle or the completion of the treatment cycles.

For multiple cycle testing, the above procedure is repeated for a set number of times. For instance, for a 5 cycle test, the above steps 1-4 are repeated five times.

#### Water Advancing Contact Angle (ACA) on Hair

- After the above procedure, water advancing contact angles (ACA) on hair was measured for each shampoo + conditioner leg after 1 cycle, 5 cycles, 10 cycles, and 20 cycles. The water advancing contact angles (ACA) on hair were measured for 5 different hairs randomly pulled from the switch in each leg tested – the average of the 5 water advancing contact angles (ACA) on hair measurements is provided below. Also, the baseline average advancing contact angles for virgin hair ( $ACA_{\text{Undamaged Virgin Hair}}$ ) and chemically damaged hair ( $ACA_{\text{Chemically Damaged Hair}}$ ) were taken as the overall average for the Clarifying shampoo only treatment (leg 1) on each of these substrates across the 1, 5, 10 and 20 treatment cycles.

- Additionally, a Hair Damage Repair Index was measured for each shampoo + conditioner leg using the water advance contact angles (ACA) on hair taken after 1 cycle, 5 cycles, 10 cycles, and 20 cycles, as described above.

Table 12: Baseline Water Advancing Contact Angles

Water Advancing Contact Angle (Baseline Undamaged Virgin Hair)	87.1	+/- 2.8 degrees
Water Advancing Contact Angle (Baseline Chemically Bleached Damaged Hair)	65.8	+/- 4.0 degrees

Table 13: Water Advancing Contact Angles (Treated Undamaged Virgin Hair)

<b>Leg</b>		<b>1 Cycle</b>	<b>5 Cycles</b>	<b>10 Cycles</b>	<b>20 Cycles</b>	<i>1 cycle Standard Deviation</i>	<i>5 cycles Standard Deviation</i>	<i>10 cycles Standard Deviation</i>	<i>20 cycles Standard Deviation</i>
1	Pantene Clarifying Shampoo	86.6	87.1	90.7	83.9	5.8	3.0	6.1	9.5
2	Dove Daily Moisture Shampoo plus Dove Daily Moisture Conditioner	103.0	100.3	96.9	97.4	4.2	2.7	4.9	4.3
3	Pantene Anti-breakage Shampoo plus Pantene Anti-breakage Conditioner	90.9	85.1	97.5	94.8	5.2	3.3	2.2	6.0
4	Pantene Expert Intensive Color Shampoo plus Pantene Repair & Protect Conditioner	96.8	107.7	95.2	101.3	10.4	3.1	10.1	4.3
5	Pantene Expert Intensive Color Shampoo plus Pantene Expert Dry Defy Conditioner	97.1	91.4	89.9	92.4	6.1	5.7	4.8	4.7
6	Example 1 Foam Shampoo plus Dove Foam Conditioner	84.7	85.1	90.1	92.8	1.8	3.4	4.4	3.1
7	Example 1 Foam Shampoo plus Example 2 Foam Conditioner	106.5	108.6	110.8	113.7	1.2	0.2	2.2	1.2
8	Example 1 Foam Shampoo plus Example 3 Foam Conditioner	105.7	108.7	108.6	117.2	0.7	0.8	0.8	2.3
9	Example 1 Foam Shampoo plus Example 4 Foam Conditioner	116.5	117.4	112.2	113.8	1.5	2.1	2.2	0.5

Table 14: Advancing Contact Angles (Treated Chemically Bleached Damaged Hair)

<b>Leg</b>		<b>1 Cycle</b>	<b>5 Cycles</b>	<b>10 Cycles</b>	<b>20 Cycles</b>	<i>1 cycle Standard Deviation</i>	<i>5 cycles Standard Deviation</i>	<i>10 cycles Standard Deviation</i>	<i>20 cycles Standard Deviation</i>
1	Pantene Clarifying Shampoo	71.0	66.3	64.6	61.4	0.9	1.2	2.2	2.6
2	Dove Daily Moisture Shampoo plus Dove Daily Moisture Conditioner	75.1	72.5	64.7	69.1	4.8	2.1	2.3	6.8
3	Pantene Anti-breakage Shampoo plus Pantene Anti-breakage Conditioner	68.7	72.9	59.2	64.1	2.1	1.1	2.1	2.9
4	Pantene Expert Intensive Color Shampoo plus Pantene Repair & Protect Conditioner	77.3	75.1	80.2	67.6	3.9	4.1	5.2	3.3
5	Pantene Expert Intensive Color Shampoo plus Pantene Expert Dry Defy Conditioner	73.7	70.3	63.2	61.1	2.0	3.0	2.0	3.2
6	Example 1 Foam Shampoo plus Dove Foam Conditioner	65.1	55.3	53.0	54.4	3.4	1.2	1.1	5.0
7	Example 1 Foam Shampoo plus Example 2 Foam Conditioner	103.0	106.7	109.8	112.9	1.1	0.3	1.4	3.6
8	Example 1 Foam Shampoo plus Example 3 Foam Conditioner	100.2	109.6	111.5	113.7	3.2	2.7	2.1	1.8
9	Example 1 Foam Shampoo plus Example 4 Foam Conditioner	116.9	113.5	119.6	120.3	2.8	1.5	1.9	3.4

Table 15: Hair Damage Repair Index

<b>Leg</b>		<b>1 Cycle</b>	<b>5 Cycles</b>	<b>10 Cycles</b>	<b>20 Cycles</b>
1	Pantene Clarifying Shampoo	0.24	0.02	-0.06	-0.21
2	Dove Daily Moisture Shampoo plus Dove Daily Moisture Conditioner	0.44	0.31	-0.05	0.15

3	Pantene Anti-breakage Shampoo plus Pantene Anti-breakage Conditioner	0.14	0.33	-0.31	-0.08
4	Pantene Expert Intensive Color Shampoo plus Pantene Repair & Protect Conditioner	0.54	0.44	0.68	0.08
5	Pantene Expert Intensive Color Shampoo plus Pantene Expert Dry Defy Conditioner	0.37	0.21	-0.12	-0.22
6	Example 1 Foam Shampoo plus Dove Foam Conditioner	-0.03	-0.49	-0.60	-0.54
7	Example 1 Foam Shampoo plus Example 2 Foam Conditioner	1.75	1.92	2.07	2.21
8	Example 1 Foam Shampoo plus Example 3 Foam Conditioner	1.62	2.06	2.15	2.25
9	Example 1 Foam Shampoo plus Example 4 Foam Conditioner	2.40	2.24	2.53	2.56

### Advancing Contact Angle Test Method

#### 5 Testing Conditions:

The method developed by Franz J. Wortmann, Gabriele Wortmann, and Erik Schulze zur Wiesche published in Langmuir 2010, 26(10), 7365–7369 was used to determine advancing water contact angles on hair fibers. A schematic is shown below.

- 10 Briefly a small segment of hair is mounted to a microbalance. The segment is perpendicularly immersed into solvents of known properties (density, surface tension, and viscosity). The microbalance determines the mass of solvent interacting with the segment. The wetting force ( $F_w$ ) is determined by multiplying the mass by the gravitational constant. Wilhelmy rod equation of state is used to convert the wetting force into contact angles. The specifics of each of these
- 15 steps are discussed below.

#### Sample Preparation of Hair Segments:

- 20  $20 \pm 2$  mm hair segments were removed from a hair within 4 cm of the tip, within 4 cm of the middle of the hair, and within 4 cm of the root end of the hair. The areas sampled were relatively straight. Straightness is defined as sufficiently free of curvature so that the segment of hair

inserted into the solvent did not buckle or deflect on the surface of the solvent during the experiment. The problem of buckling of the hydrophobic fibers in the advancing mode have been reported and discussed by Lodge, R.A. and Bushan, B. *J. Appl. Polym. Sci.* 2006, **102**, 5255-5265. If the segment buckled to the point that the segment did not penetrate the surface of the solvent and immerse into the bulk of the solvent, that segment was replaced.

#### Measurement Principle:

Measurements were conducted with the Krüss Tensiometer K100 SF (Krüss, Hamburg, Germany – Laboratory Desktop build 3.2.2.3064 with contact angle module). Laboratory conditions were 23±1°C and 50±4% relative humidity. To measure the dynamic wetting force ( $F_w$ ), the hair is suspended underneath a microbalance with its root end pointing downward perpendicularly to the surface of the solvent (water) below. Either the hair segment was attached via a clip or double sided sticky tape to ensure the hair is perpendicular to the water surface. The mass of the hair segment was determined. Diameter of hair was optically measured with camera with sufficient magnification to enable the determination of the diameter through calibrated distance measurements. Initial immersion speeds were 6 mm/min until the surface of the fluid contacted the root end of the hair segment. Upon detection of the surface, the segment was inserted 0.2 mm. Measurement immersion speed was reduced to 0.2mm/min after surface detection and immersion to 0.2 mm. The mass of the segment was then determined after 1 s delay. After the segment was immersed an additional 100 µm the immersion was stopped for 1 s to determine the mass ( $m$ ) of the segment. Immersion continued until 0.5 cm length of the hair segment was measured. Wetting forces ( $F_w$ ) was determined by multiplying the mass by the gravitational constant ( $g$ ). These measurements constituted advancing wetting forces.

$$F_w = mg$$

$F_w$  was converted to contact angle ( $\theta$ ) with the Wilhelmy rod equation of state:

$$F_w = L\sigma_L \cos \theta$$

Wherein  $L$  is the wetted perimeter of the fiber at the air/liquid interface or the circumference of the hair,

$\sigma_L$  the surface tension of the liquid, and

$\theta$  the contact angle

L was determined from the diameter (d) of the hair.

$$L = \pi d$$

5

#### Hair Damage Repair Index Test Method

The Hair Damage Repair Index is based on the advancing contact angle (ACA) on hair of the baseline undamaged virgin hair, the baseline chemically damaged hair, and the chemically damaged hair after the treatment cycle.

10

*Hair Damage Repair Index<sub>Treatment</sub>*

$$= \frac{ACA_{\text{Chemically Damaged Hair after Treatment}} - ACA_{\text{Chemically Damaged Hair}}}{ACA_{\text{Undamaged Virgin Hair}} - ACA_{\text{Chemically Damaged Hair}}}$$

15

Although the advancing contact angles (ACA) on hair measurements were taken for the undamaged virgin hair after the treatment legs (and are provided above), these values are not used in the Hair Damage Repair Index calculation. However, they are relevant for comparison purposes.

#### Atomic Force Microscopy and Deposited Silicone Layer Thickness

A non-depositing “clarifying” shampoo was employed (Pantene Pro-V Purifying Shampoo) in the below examples that was void of high melting point fatty compounds and conditioning agents.

The Table 16 aerosol conditioner compositions were prepared by weighing distilled water and the oil (aminosilicone) emulsions into a stainless steel beaker. The beaker is placed in a water bath on a hot plate while mixing with overhead mixer at 100 to 150 rpm. If fatty alcohols are present in the formula, the cetyl alcohol and stearyl alcohol are added and the mixture is heated to 70-75C. Cetyltrimethylammonium chloride is then added and mixing speed is increased to 250-350 rpm due to viscosity increase. When the materials are all heated thoroughly and homogenous, the heating is stopped while the mixture is continued to stir. The batch is cooled to 35C by removing

the hot water from the water bath and replacing with cold water. The perfume and Kathon are added and with continued stirring for ~ 10 minutes. For foaming, the batch is transferred to appropriate container and propellant Aeron-46 is added.

**Table 16**

Raw Material	Ex 1	Ex 2
Aminosilicone <sup>1</sup>	12	12
Perfume	2.4	3.0
Cetyltrimethylammonium Chloride		2.5
Cetyl Alcohol		3.0
Stearyl Alcohol		3.0
Preservative (Kathon)	0.03	0.03
Propellant (Aeron-46)	4.0	4.0
Water (q.s.)		
Weight ratio of oil to high melting point fatty compounds	100:0	67:33

<sup>1</sup> Silsoft 253 (20% active) available from Momentive

Each of the Table 16 conditioners was treated onto virgin brown hair switches as part of a regimen with Pantene Pro-V Clarifying Shampoo for up to 6 or 7 treatment cycles. As regimen controls, the Pantene Pro-V Clarifying Shampoo was combined with Pantene Silky Smooth Care Conditioner and Pantene Pro-V Anti-Breakage Conditioner. These conditioners are known to have aminosilicone contents of about 2.5% and 4.0% respectively, and a total high melting point fatty compounds (cetyl and stearyl alcohols) content of 5.20% and 5.20%, respectively for a weight ratio of silicone to high melting point fatty compounds in the formula of 43.5:56.5 and 32.5:67.5, respectively.

**Multiple Cycle Shampoo plus Conditioner treatments:**

1. Six 4 gram, 8 inch hair switches (virgin brown hair) are wet with 100 degrees Fahrenheit water at a sink (bound on root-ends with glue/tape and hanging on metal holder) with a shower head fixture (flow rate is 1.5 gallons per minute) for 15 to 20 seconds.
2. Liquid shampoos are applied at 0.1grams of product per gram of hair (e.g., Pantene Pro-V Purifying Shampoo) via a syringe and milked/scrubbed for 30 seconds followed by a 30 seconds shower head rinse (with gentle manipulation at top of switch to ensure uniform rinsing).



3. Liquid conditioners are applied at 0.1 grams of product per gram of hair (e.g., Pantene Anti-breakage Conditioner etc.) via a syringe (weighed on weigh scale) evenly over the hair switch and milked/scrubbed for 30 seconds followed by a 30 seconds shower rinse (with gentle manipulation at top of switch to ensure uniform rinsing). Concentrated liquid foam conditioners are applied at 0.033 grams of product per gram of hair with a spatula (foam is dispensed in weigh boat and applied weight recorded) and following the same application procedure.
4. The hair is then dried in a heat box set at 60C for ~45 minutes or until mostly dry before starting the next treatment cycle or the completion of the treatment cycles.

For multiple cycle testing, the above procedure is repeated for a set number of times. For instance, for a six cycle test, the above steps 1-4 are repeated six times.

#### AFM Deposit Thickness & Deposition Data

	Pantene Clarifying Shampoo Only	Pantene Clarifying Shampoo plus Pantene Silky Smooth Care Conditioner	Pantene Clarifying Shampoo plus Pantene Anti-breakage Conditioner	Clarifying Shampoo plus Ex 1 Conditioner	Clarifying Shampoo plus Ex 2 Conditioner
No. of Cycles	7	7	6	6	6
Number of AFM Images	2	2	3	3	4
Number of AFM Points	788	777	710	544	611
Average Deposit Thickness (nm)	5.3	59.8	32.8	15.4	15.1
Standard Deviation of Deposit Thickness (nm)	1.76	87.9	55.0	9.5	9.8
Minimum Deposit Thickness (nm)	2.7	6.8	3.6	5.5	3.8
Maximum Deposit Thickness (nm)	16.6	613.2	439.4	80.2	100.1
Coefficient of Variation of Deposition Thickness	33%	147%	168%	62%	65%

Thickness of deposition Range (nm)	13.9	606.4	435.8	74.7	96.3
Oil (Aminosilicone) Deposition (ppm)	--	1763	1557 +/- 844	1341 +/- 449	1546 +/- 67
Normalized Thickness of Deposition (nm/ppm Oil)	--	0.034	0.021	0.011	0.010
Normalized Coefficient of Variation of Deposition Thickness (%/ppm Oil)	--	0.083	0.108	0.046	0.015
Normalized Maximum thickness of deposition (nm/ppm Oil)	--	0.350	0.282	0.060	0.065

### Hair Conditioner Oil to High Melting Point Fatty Compound Ratios

	Pantene Clarifying Shampoo plus Pantene Silky Smooth Care Conditioner	Pantene Clarifying Shampoo plus Pantene Anti-breakage Conditioner	Clarifying Shampoo plus Ex 1 Conditioner	Clarifying Shampoo plus Ex 2 Conditioner
% Oil (Aminosilicone)	4	2.5	12	12
% High Melting Point Fatty Compound (cetyl and stearyl alcohols)	5.2	5.2	0	6
Oil to High melting point fatty compound ratio	43.5:56.5	32.5:67.5	100:0	67:33
Product Density (g/cm <sup>3</sup> )	1.0	1.0	0.06	0.06
Product Dosage (grams per grams of hair)	0.10	0.10	0.033	0.033
Average product dosage	10g	10g	3.3g	3.3g

The AFM data and images after 6-7 treatment cycles on virgin hair demonstrates that the concentrated conditioners described herein provide surface deposits with improved morphology (thinner, smoother and more even deposition) versus the liquid control regimen (irregular deposits that are thicker, not smooth and lacking even deposition). Additionally, the AFM was able to quantify the thickness of the deposits and demonstrating the inventive concentrated conditioners described herein to provide:

- Significantly thinner deposits with lower average thickness of deposits (averages of 15.4 +/- 9.5 nanometers and 15.1 +/- 9.8 nanometers) versus the currently marketed liquid control conditioner (32.8 +/- 55 nanometers).
- More uniform deposits with very low thickness coefficient of variations (62% and 65%) versus the currently marketed liquid control conditioner (168%). The coefficient of variation (CV) is defined as the ratio of the standard deviation to the mean and is expressed as a percentage.
- More uniform deposits with a much lower range in the thickness of the deposits (74.7 nanometers and 96.3 nanometers) versus the currently marketed liquid control conditioner (435.8 nanometers). The range is defined as the difference between the largest and smallest measured thickness values.
- Up to 70% lower maximum thickness (80.2 nanometers and 100.1 nanometers) versus the currently marketed liquid control conditioner (439.4 nanometers).

Moreover, combining the AFM data with the measured deposition data demonstrates the inventive concentrated conditioners described herein to provide:

- 50% lower normalized thickness of deposition.
- Up to 80% lower normalized coefficient of variation of deposition thickness.
- Greater than 75% lower normalized maximum thickness of deposition.

#### Atomic Force Microscopy Test Method (conducted after 6-7 treatment cycles)

Samples are prepared for AFM analysis by selecting 2-4 hairs from those provided and adhering an approximately 1 inch long section to a hard substrate (e.g., glass microscope slide) with quick curing epoxy; the analysis region was approximately the middle of the hair. The intention was to secure the sample under a small amount of tension such that it did not move under the influence of the AFM probe. Images were collected in tapping (intermittent contact) mode with a Field of View (FOV) of 20 x 20 or 40 x 20 micrometers at a lateral resolution of about 40-80 nanometers.

The depth or thickness of a layer of soft material lying on top of a harder substrate can be determined from Deflection-Displacement curves collected with an AFM. For this measurement the stiffness of the cantilever should be greater than that of the soft film and should be less than that of the underlying substrate. In analyzing the deflection-displacement curve, the relative distance that the cantilever travels between the point where the cantilever snaps into contact with the soft material and the point where the cantilever begins to deflect strongly from contact with the substrate is taken to be the thickness of the overlying layer of soft material. This applies to the measurement of layer thickness at a single point; this measurement can be extended to the determination of layer thickness over an area by collecting an array of deflection-displacement curves with spacing between measurement locations designed to give the desired spatial resolution. Deflection-displacement curves were collected over the same areas as imaged by AFM described above. Arrays of individual deflection-displacement curves were uniformly distributed over the FOV, usually at a spacing of ~1 micrometer excluding imaged area edges. About 500-800 hundred individual curves (Number of AFM Points) were collected over areas of 2-4 AFM images (Number of AFM Images) and Average, Standard Deviation of, Minimum, and Maximum Deposit Thickness were found for each type of measured samples. An example suitable instrument is the Asylum Research model MFP-3D; example suitable cantilever is the Olympus AC160-TS with a nominal spring constant of 42 N/m.

Deposition Data Test Method (conducted after 6-7 treatment cycles):

Silicone deposition is determined by digestion of the hair followed by an analysis with a quantitative elemental technique such as ICP for total silicon and converting to silicone based on the % of silicon in the silicone by weight. The total deposition may be determined by the sum of separate deposition measurements. The separate deposition measurements may include but are not limited to: fatty alcohols, EGDS, quaternized agents and silicone. Typically these measurements involve extracting the hair then separating the ingredients of interest with chromatography and quantifying with an external calibration based on test solution concentration using an appropriate detector.

ICP-OES silicone hair digestion method: Hair samples treated with different products are submitted as balls of hair with an average sample size of 0.1 g. These hair samples are then digested using a single reaction chamber microwave digestion system (Milestone Inc., Shelton, CT) using a 6:1 HNO<sub>3</sub>:H<sub>2</sub>O<sub>2</sub> mixture and an aliquot of methyl isobutyl ketone (MIBK) in Teflon

digestion vessels. A gentle digestion program with a ramp to 95°C and a manual vent after cooling below 30°C is used to facilitate retention of silicon. After dilution to volume, the samples are run against an inorganic silicon calibration curve produced on an Optima 8300 ICP-OES system (Perkin Elmer, Waltham, Massachusetts) run in the axial mode. The silicon values determined are converted to a concentration of silicone polymer-equivalents deposited on the hair sample using the theoretical silicon concentration of the polymer provided by the manufacturer. An untreated hair sample is analyzed to determine the background concentration of silicon to allow correction if needed. Another untreated hair sample is spiked with a known amount of polymer and analyzed to ensure recovery of the polymer and verify the analysis.

### **Silicone Layer Targeted Deposition Index and % Targeted Deposition**

#### **Concentrated Hair Conditioner Examples**

The concentrated hair conditioner examples in Table 16 were made by mixing together water, the aminosilicone (oil) emulsion, and EDTA in a beaker. The mixture was heated to 65 degrees Celsius with continuous and thorough mixing via an overhead mixer (covered in foil to mitigate evaporation). Stearyl alcohol and cetyl alcohol are then added with continued heating to 85 degrees Celsius while stirring. The behentrimethylammonium methosulfate is then added with continued mixing for 10 minutes at a temperature of 80 degrees Celsius. The mixture is then allowed to cool with continued mixing to below 30 degrees Celsius. The benzyl alcohol, perfume, Kathon and citric acid are then added and mixed for 10 minutes to complete the batch.

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Table 17

Raw Material	Ex 1	Ex 2	Ex 3	Ex 4	Ex 5
Aminopropylaminoethylpolysiloxane <sup>1</sup>	8	8	8	8	8
Perfume	2.0	2.0	2.0	2.0	2.0
Behentrimethylammonium methosulfate <sup>2</sup>	28.69	18.72	12.53	5.33	1.06
Cetyl Alcohol	4.64	3.03	2.03	0.86	0.17
Stearyl Alcohol	11.60	7.57	5.07	2.15	0.43
Benzyl Alcohol	0.4	0.4	0.4	0.4	0.4
Citric Acid	0.02	0.02	0.02	0.02	0.02
Preservative (Kathon)	0.03	0.03	0.03	0.03	0.03
Tetrasodium EDTA	0.13	0.13	0.13	0.13	0.13
Water (q.s.)					

Quat to Fatty Alcohol Ratio	1.77	1.77	1.77	1.77	1.77
Stearyl Alcohol to Cetyl Alcohol Ratio	2.50	2.50	2.50	2.50	2.50
Weight Ratio of oil to high melting point fatty compounds	0.49	0.75	1.13	2.66	13.33
Proportion of oil to high melting point fatty compounds	33:67	43:57	53:47	73:27	93:07
Silicone to (High Melting Point + Cationic Surfactant) Weight Ratio	0.18	0.27	0.41	0.96	4.82

1 Aminosilicone micro-emulsion (Silsoft 253), available from Momenitive

2 BTMS (Genamin BTMS, 77-85% active), available from Clariant

The concentrated hair conditioner examples in Table 17 were benchmarked versus several currently marketed hair conditioners that were purchased from a retail store including Pantene Color Preserve Shine Conditioner, Pantene Anti-breakage Conditioner, and Pantene Sheer Volume Conditioner. The above conditioners were treated as the 2<sup>nd</sup> step of a 2 step system with currently marketed Pantene Clarifying Shampoo.

In the following data, the hair was chemically damaged with a hair bleaching composition comprising 5.5% hydrogen peroxide, 2% ammonium hydroxide, and 0.2% EDTA. The hair was submerged within this hair bleaching composition for a period of 35 minutes followed by thorough rinsing. The hair was then dried while brushing continuously for 3 minutes (1.5 minutes per side). However, the measured damage repair index of the present invention is normalized and thereby applicable to any degree of consumer relevant hair damage including subjecting the hair to a bleaching composition for shorter (down to 1 minute) and longer time periods (up to several hours) provided statistically meaningful differences can be measured between the starting undamaged hair and damaged hair substrates.

The hair substrates employed for this study were 4 gram, 8 inch brown virgin (general population) hair tresses available from International Hair Importers & Products (Glendale, NY, inhip@aol.com).

#### Shampoo plus Conditioner treatments:

The above Pantene Clarifying Shampoo and conditioner pairings were treated onto virgin brown hair and chemically bleached damaged hair through 20 hair treatment cycles:

5. Six 4 gram, 8 inch hair switches (virgin brown hair or chemically bleached damaged hair) are wet with 100 degrees Fahrenheit water at a sink (bound on root-ends with glue/tape and hanging on metal holder) with a shower head fixture (flow rate is 1.5 gallons per minute) for 15 to 20 seconds.
6. Liquid shampoos are applied at 0.1grams of product per gram of hair (e.g., Pantene Pro-V Clarifying Shampoo) via a syringe and milked/scrubbed for 30 seconds followed by a 30 seconds shower head rinse (with gentle manipulation at top of switch to ensure uniform rinsing).
7. Liquid conditioners are applied at 0.1 grams of product per gram of hair (e.g., Pantene Anti-breakage Conditioner etc.) via a syringe (weighed on weigh scale) evenly over the hair switch and milked/scrubbed for 30 seconds followed by a 30 seconds shower rinse (with gentle manipulation at top of switch to ensure uniform rinsing). The concentrated liquid foam conditioners of the present invention are applied at 0.033 grams of product per gram of hair with a spatula (foam is dispensed in weigh boat and applied weight recorded) and following the same application procedure.
8. The hair is then dried in a heat box set at 60C for ~45 minutes or until dry.

Silicone Deposition Data and Targeted Deposition Ratio:

Silicone deposition is determined by digestion of the hair followed by an analysis with a quantitative elemental technique such as ICP for total silicon and converting to silicone based on the % of silicon in the silicone by weight. The total deposition may be determined by the sum of separate deposition measurements. The separate deposition measurements may include but are not limited to: fatty alcohols, EGDS, quaternized agents and silicone. Typically these measurements involve extracting the hair then separating the ingredients of interest with chromatography and quantifying with an external calibration based on test solution concentration.

ICP-OES silicone hair digestion method: Hair samples treated with different products are submitted as balls of hair with an average sample size of 0.1 g. These hair samples are then digested using a single reaction chamber microwave digestion system (Milestone Inc., Shelton, CT) using a 6:1 HNO<sub>3</sub>:H<sub>2</sub>O<sub>2</sub> mixture and an aliquot of methyl isobutyl ketone (MIBK) in Teflon digestion vessels. A gentle digestion program with a ramp to 95°C and a manual vent after cooling below 30°C is used to facilitate retention of silicon. After dilution to volume, the samples are run against an inorganic silicon calibration curve produced on an Optima 8300 ICP-OES system (Perkin Elmer, Waltham, Massachusetts) run in the axial mode. The silicon values

determined are converted to a concentration of silicone polymer-equivalents deposited on the hair sample, reported in ppm, using the theoretical silicon concentration of the polymer provided by the manufacturer. An untreated hair sample is analyzed to determine the background concentration of silicon to allow correction if needed. Another untreated hair sample is spiked with a known amount of polymer and analyzed to ensure recovery of the polymer and verify the analysis.

The resulting deposition data is given in Table 18.

Table 18

	<b>Silicone Deposition (ppm)-Undamaged Virgin Hair</b>	<b>Silicone Deposition (ppm)-Chemically Damaged Hair</b>
Pantene Color Preserve Shine Conditioner	196 +/- 28	128 +/- 27
Pantene Antibreakage Conditioner	273 +/- 23	202 +/- 38
Pantene Sheer Volume Conditioner	282 +/- 10	197 +/- 23
Example 1 Concentrated Conditioner	407 +/- 30	308 +/- 26
Example 2 Concentrated Conditioner	581 +/- 101	447 +/- 37
Example 3 Concentrated Conditioner	510 +/- 20	572 +/- 39
Example 4 Concentrated Conditioner	787 +/- 55	879 +/- 149
Example 5 Concentrated Conditioner	1120 +/- 29	1380 +/- 294

From this deposition data, a Targeted Deposition Delta (Chemically Damaged Deposition minus Undamaged Virgin Hair Deposition), a % Targeted Deposition (Targeted Deposition Delta divided by Undamaged Virgin Hair Deposition x 100%), and Targeted Deposition Index (Chemically Damaged Deposition divided by Undamaged Virgin Hair Deposition) can be computed as given in Table 19.

Table 19

	<b>Targeted Deposition Delta (Bleached - Virgin)</b>	<b>% Targeted Deposition (Delta/Virgin x 100%)</b>	<b>Targeted Deposition Index</b>
Pantene Color Preserve Shine Conditioner	-67.3	-34%	0.66
Pantene Antibreakage Conditioner	-70.9	-26%	0.74
Pantene Sheer Volume Conditioner	-85.0	-30%	0.70
Example 1 Concentrated Conditioner	-99.1	-24%	0.76
Example 2 Concentrated Conditioner	-133.2	-23%	0.77
Example 3 Concentrated Conditioner	61.4	12%	1.12
Example 4 Concentrated Conditioner	92.0	12%	1.12
Example 5 Concentrated Conditioner	260.0	23%	1.23



**Additional Examples/Combinations**

A. A method of treating hair, the method comprising:

a. providing a concentrated hair care composition in a foam dispenser, wherein the concentrated hair care composition comprises:

i. from about 3% to about 25% of one or more silicones, by weight of the concentrated hair care composition;

ii. less than 5% high melting point fatty compounds, by weight of the concentrated hair care composition;

iii. from about 60% to about 90% water, by weight of the concentrated hair care composition; and

iv. from about 0.5% to about 7% perfume, by weight of the concentrated hair care composition;

wherein the concentrated hair care composition has a silicone to high melting point fatty compound weight ratio of from about 100:0 to about 50:50;

wherein the concentrated hair care composition has a silicone to perfume weight ratio of from about 98:2 to about 50:50;

b. dispensing the concentrated hair care composition from the foam dispenser as a foam, wherein the foam has a density of from about 0.025 g/cm<sup>3</sup> to about 0.40 g/cm<sup>3</sup>;

c. applying the foam to the hair, wherein the one or more silicones are deposited onto the hair when applying the foam to the hair; and

d. rinsing the foam from the hair;

wherein the one or more silicones forms a silicone layer on the hair; and

wherein the silicone layer has a Targeted Deposition Index of from about 0.8 to about 1.6.

B. The method of paragraph A, wherein the Targeted Deposition Index is from about 0.9 to about 1.5.

C. The method of any one of paragraphs A or B, wherein the Targeted Deposition Index is from about 1.0 to about 1.4.

- D. The method of any one of paragraphs A-C, wherein the Targeted Deposition Index is from about 1.05 to about 1.35.
- 5 E. The method of any one of paragraphs A-D, wherein the Targeted Deposition Index is from about 1.10 to about 1.30.
- F. The method of any one of paragraphs A-E, wherein the Targeted Deposition Index is from about 0.85 to about 1.30.
- 10 G. The method of any one of paragraphs A-F, wherein the Targeted Deposition Index is from about 0.9 to about 1.30.
- H. The method of any one of paragraphs A-G, wherein the Targeted Deposition Index is from about 0.95 to about 1.30.
- 15 I. The method of any one of paragraphs A-H, wherein the Targeted Deposition Index is from about 1.0 to about 1.30.
- J. The method of any one of paragraphs A-I, wherein the Targeted Deposition Index is from about 1.10 to about 1.30.
- 20 K. The method of any one of paragraphs A-J, wherein the Targeted Deposition Index is from about 1.15 to about 1.30.
- L. The method of any one of paragraphs A-K, wherein the concentrated hair care composition has a % Targeted Deposition of from about -20% to about 50%.
- 25 M. The method of any one of paragraphs A-L, wherein the concentrated hair care composition has a % Targeted Deposition of from about -10% to about 40%.
- 30 N. The method of any one of paragraphs A-M, wherein the concentrated hair care composition has a % Targeted Deposition of from about 10% to about 25%.

- O. The method according to any one of paragraphs A-N, wherein the silicone to high melting point fatty compound weight ratio is
- P. The method according to any one of paragraphs A-O, wherein the concentrated hair care composition has a silicone to (high melting point fatty compound plus cationic surfactant) weight ratio of from about 0.30 to about 10.
- Q. The method according to any one of paragraphs A-P, wherein the silicone is a polar functional silicone.
- R. The method according to any one of paragraphs A-Q, wherein the polar functional silicone is selected from the group consisting of aminosilicones, pendant quaternary ammonium silicones, terminal quaternary ammonium silicones, amino polyalkylene oxide silicones, quaternary ammonium polyalkylene oxide silicones, amino morpholino silicones, and mixtures thereof.
- S. A foam dispenser comprising a concentrated hair care composition, wherein the concentrated hair care composition comprises:
- from about 3% to about 25% of one or more silicones, by weight of the concentrated hair care composition;
  - less than 5% high melting point fatty compounds, by weight of the concentrated hair care composition;
  - from about 60% to about 90% water, by weight of the concentrated hair care composition; and
  - from about 0.5% to about 7% perfume, by weight of the concentrated hair care composition;
- wherein the concentrated hair care composition is a rinse-off concentrated hair care composition;
- wherein the concentrated hair care composition has a silicone to high melting point fatty compound weight ratio of from about 100:0 to about 50:50;
- wherein the concentrated hair care composition has a silicone to perfume weight ratio of from about 98:2 to about 50:50;

wherein the foam has a density of from about 0.025 g/cm<sup>3</sup> to about 0.40 g/cm<sup>3</sup>; wherein the concentrated hair care composition deposits a silicone layer onto hair;  
wherein the silicone layer has a Targeted Deposition Index of from about 0.8 to about 1.6.

T. The foam dispenser of paragraph S, wherein the concentrated hair care composition has a % Targeted Deposition of from about -20% to about 50%.

The dimensions and values disclosed herein are not to be understood as being strictly limited to the exact numerical values recited. Instead, unless otherwise specified, each such dimension is intended to mean both the recited value and a functionally equivalent range surrounding that value. For example, a dimension disclosed as “40 mm” is intended to mean “about 40 mm.”

Every document cited herein, including any cross referenced or related patent or application and any patent application or patent to which this application claims priority or benefit thereof, is hereby incorporated herein by reference in its entirety unless expressly excluded or otherwise limited. The citation of any document is not an admission that it is prior art with respect to any invention disclosed or claimed herein or that it alone, or in any combination with any other reference or references, teaches, suggests or discloses any such invention. Further, to the extent that any meaning or definition of a term in this document conflicts with any meaning or definition of the same term in a document incorporated by reference, the meaning or definition assigned to that term in this document shall govern.

While particular embodiments of the present invention have been illustrated and described, it would be obvious to those skilled in the art that various other changes and modifications can be made without departing from the spirit and scope of the invention. It is therefore intended to cover in the appended claims all such changes and modifications that are within the scope of this invention.

## CLAIMS

What is claimed is:

- 1) A method of treating hair, the method comprising:
  - a. providing a concentrated hair care composition in a foam dispenser, wherein the concentrated hair care composition comprises:
    - i. from 3% to 25% of one or more silicones, by weight of the concentrated hair care composition;
    - ii. less than 5% high melting point fatty compounds, by weight of the concentrated hair care composition;
    - iii. from 60% to 90% water, by weight of the concentrated hair care composition; and
    - iv. from 0.5% to 7% perfume, by weight of the concentrated hair care composition;wherein the concentrated hair care composition has a silicone to high melting point fatty compound weight ratio of from 100:0 to 50:50;  
wherein the concentrated hair care composition has a silicone to perfume weight ratio of from 98:2 to 50:50;
  - b. dispensing the concentrated hair care composition from the foam dispenser as a foam, wherein the foam has a density of from 0.025 g/cm<sup>3</sup> to 0.40 g/cm<sup>3</sup>;
  - c. applying the foam to the hair, wherein the one or more silicones are deposited onto the hair when applying the foam to the hair; and
  - d. rinsing the foam from the hair;  
wherein the one or more silicones forms a silicone layer on the hair; and  
wherein the silicone layer has a Targeted Deposition Index of from 0.8 to 1.6.
- 2) The method of Claim 1, wherein the Targeted Deposition Index is from 0.9 to 1.5.
- 3) The method of Claim 1 or 2, wherein the Targeted Deposition Index is from 1.05 to 1.35.
- 4) The method of any preceding claim, wherein the Targeted Deposition Index is from 0.85 to 1.30.

- 5) The method of any preceding claim, wherein the Targeted Deposition Index is from 0.95 to 1.30.
- 6) The method of any preceding claim, wherein the Targeted Deposition Index is from 1.10 to 1.30.
- 7) The method of any preceding claim, wherein the concentrated hair care composition has a % Targeted Deposition of from -20% to 50%.
- 8) The method of any preceding claim, wherein the concentrated hair care composition has a % Targeted Deposition of from -10% to 40%.
- 9) The method of any preceding claim, wherein the concentrated hair care composition has a % Targeted Deposition of from 10% to 25%.
- 10) The method of any preceding claim, wherein the silicone to high melting point fatty compound weight ratio is from about 100:0 to about 60:40.
- 11) The method of any preceding claim, wherein the concentrated hair care composition has a silicone to (high melting point fatty compound plus cationic surfactant) weight ratio of from 0.30 to 10.
- 12) The method of any preceding claim, wherein the silicone is a polar functional silicone.
- 13) The method of Claim 12, wherein the polar functional silicone is selected from the group consisting of aminosilicones, pendant quaternary ammonium silicones, terminal quaternary ammonium silicones, amino polyalkylene oxide silicones, quaternary ammonium polyalkylene oxide silicones, amino morpholino silicones, and mixtures thereof.

- 14) A foam dispenser comprising a concentrated hair care composition, wherein the concentrated hair care composition comprises:
- i. from 3% to 25% of one or more silicones, by weight of the concentrated hair care composition;
  - ii. less than 5% high melting point fatty compounds, by weight of the concentrated hair care composition;
  - iii. from 60% to 90% water, by weight of the concentrated hair care composition; and
  - iv. from 0.5% to 7% perfume, by weight of the concentrated hair care composition;
- wherein the concentrated hair care composition is a rinse-off concentrated hair care composition;
- wherein the concentrated hair care composition has a silicone to high melting point fatty compound weight ratio of from 100:0 to 50:50;
- wherein the concentrated hair care composition has a silicone to perfume weight ratio of from 98:2 to 50:50;
- wherein the foam has a density of from 0.025 g/cm<sup>3</sup> to 0.40 g/cm<sup>3</sup>; wherein the concentrated hair care composition deposits a silicone layer onto hair;
- wherein the silicone layer has a Targeted Deposition Index of from 0.8 to 1.6.
- 15) The foam dispenser of Claim 14, wherein the concentrated hair care composition has a % Targeted Deposition of from -20% to 50%.

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FIG. 1

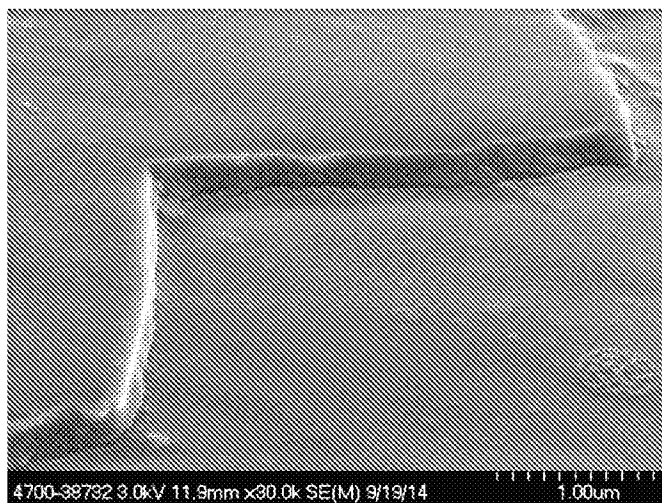


FIG. 2



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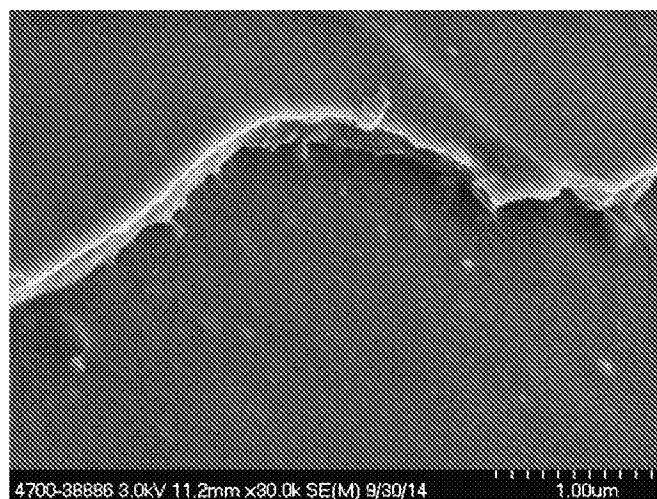


FIG. 3

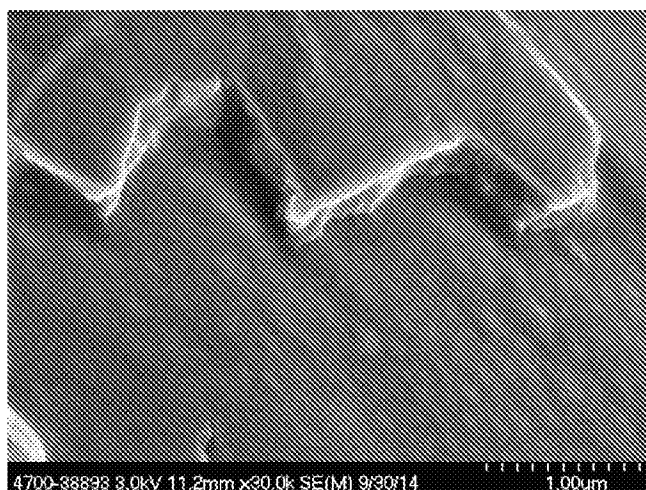


FIG. 4

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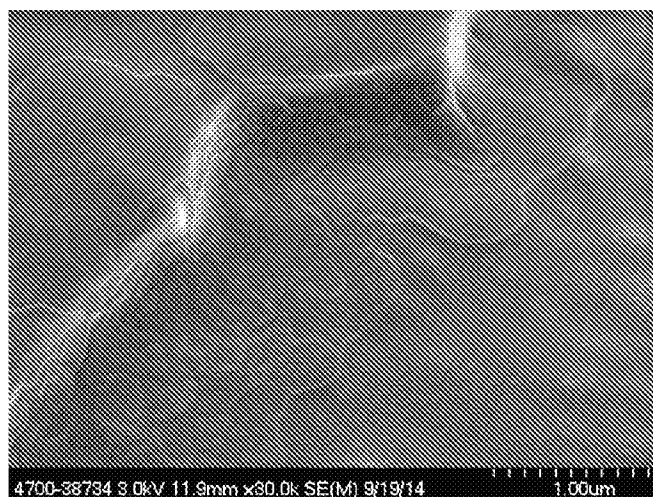


FIG. 5

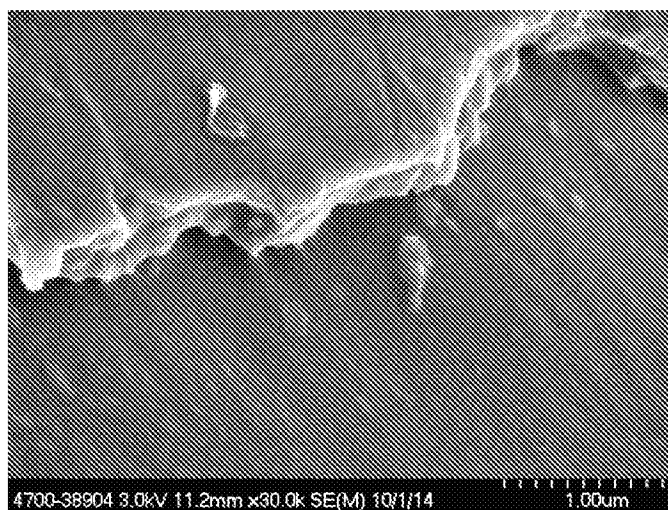


FIG. 6

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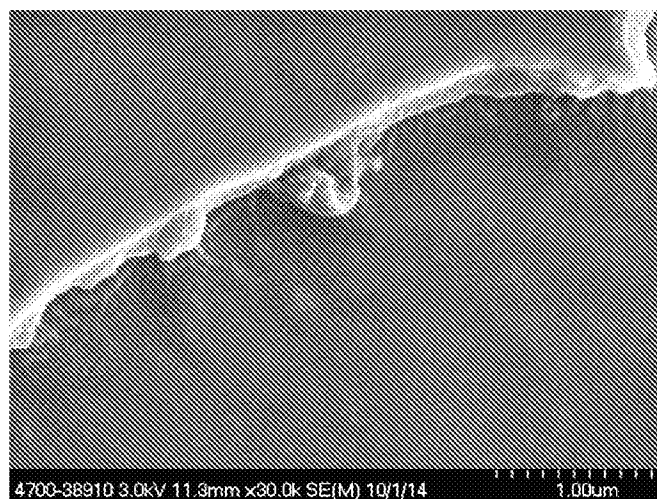


FIG. 7

## INTERNATIONAL SEARCH REPORT

International application No  
PCT/US2017/028473

## A. CLASSIFICATION OF SUBJECT MATTER

INV. A61K8/898 A61K8/04 A61Q5/12  
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

A61Q 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 practicable, search terms used)

EPO-Internal, BIOSIS, Sequence Search, EMBASE, EMBL, INSPEC, CHEM ABS Data, SCISEARCH, WPI Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2015/359725 A1 (GLENN JR ROBERT WAYNE [US] ET AL) 17 December 2015 (2015-12-17) paragraphs [0006], [0007], [0031] - [0074], [0145], [0172]; claims; figures 1-6; examples 1-3,8-13; tables 2-8 -----	1-15
X	US 2015/359726 A1 (GLENN JR ROBERT WAYNE [US] ET AL) 17 December 2015 (2015-12-17) paragraphs [0006], [0007], [0030] - [0095], [0158], [0162]; claims; examples 6-11 -----	1-15
A	US 2015/359727 A1 (GLENN JR ROBERT WAYNE [US] ET AL) 17 December 2015 (2015-12-17) the whole document -----	1-15
A	US 2015/359728 A1 (GLENN JR ROBERT WAYNE [US] ET AL) 17 December 2015 (2015-12-17) the whole document -----	1-15



Further documents are listed in the continuation of Box C.



See patent family annex.

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Date of the actual completion of the international search

21 June 2017

Date of mailing of the international search report

29/06/2017

Name and mailing address of the ISA/

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040,  
Fax: (+31-70) 340-3016

Authorized officer

Steffen, Pierre

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2017/028473

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