(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization

International Bureau





(10) International Publication Number WO 2014/020081 A2

(43) International Publication Date 6 February 2014 (06.02.2014)

(51) International Patent Classification: A61K 8/81 (2006.01) A61K 8/04 (2006.01) A61Q 5/12 (2006.01)

(21) International Application Number:

PCT/EP2013/066124

(22) International Filing Date:

31 July 2013 (31.07.2013)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

12179310.3 3 August 2012 (03.08.2012)

EP

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- (81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.
- (84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:

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

Published:

without international search report and to be republished upon receipt of that report (Rule 48.2(g))



(54) Title: COMPOSITION

(57) Abstract: Conditioning composition obtainable by making a conditioning gel phase and then adding polyelectrolyte complex (PEC) to the gel phase, wherein the conditioning gel phase comprises a cationic surfactant and/or a fatty alcohol and wherein the composition comprises from 0 to 2% by weight anionic surfactant.

COMPOSITION

The present invention relates to improved conditioning compositions.

The combination of polyquat-28 and methylvinylether/maleic acid copolymer (PVMMA) at particular ratios produce a polyelectrolyte complex (PEC) that repairs split-end damage (U.S. Patent Application Publication Nos. 2005/0089494 and 2006/0251603, both assigned to ISP Investments Inc.). However, within the context of hair care formulations with conditioning actives, the stability of PECs may be compromised due to the presence of other ingredients, especially charged compounds and polymers that could disrupt the PEC structure. Thus, the usefulness of PECs may be limited by their compatibility with other ingredients that may be desirable to include in a hair care composition. Since multiple properties are often desired in hair care compositions, it would be desirable to formulate hair care compositions that include stable combinations of PECs with other hair care ingredients that provide additional desirable properties.

WO2012/054243 (Alberto Culver) discloses hair compositions with PEC. The document discloses means for formulating conditioning compositions with PEC. However, the formulation technique described therein is not suitable for conditioning compositions which comprise a conditioning gel phase which is in lamellar phase.

It would therefore be desirable to formulate a stable hair care composition that includes intact PECs in combination with a conditioning gel phase.

The present invention also provides methods of using the compositions described herein, including a method of conditioning a hair fiber and a method of repairing a split-end of a hair fiber.

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Despite the prior art there remains a need for hair conditioning compositions with PEC.

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Accordingly, the present invention provides a composition according to claim 1.

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We have surprisingly found that PEC can be stably incorporated into hair conditioning compositions with lamellar conditioning gel phase if the PEC is added to the composition after formation of the conditioning gel phase and not before it. This is in contrast to what is disclosed in the prior art which suggests that further thickening materials such as PQ-37 or PEG are required to maintain structure. However, the compositions disclosed in the prior art do not refer to compositions with conditioning gel phases and instead relate solely to compositions comprising materials with conditioning capability without the need for lamellar phase.

The PEC is thus added to the conditioning gel phase and not formed within the gel phase and is not prepared separately and the gel phase added to it.

The composition according to the invention is a conditioning composition. Further, it exhibits a conditioning capability by way of a conditioning gel phase which is in lamellar phase.

Preferably, the composition is a dedicated conditioning composition and as a whole comprises from 0 to 2% wt. anionic surfactant and more preferably from 0 to 1% wt. anionic surfactant.

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Preferably, the composition as a whole comprises substantially no anionic surfactant.

Such a conditioning gel phase is common in the art and means an arrangement of cationic surfactant and fatty materials, preferably fatty alcohols, in water which are WO 2014/020081

capable of conditioning hair. Silicones are also be present in the formulation but are typically not part of the conditioning gel phase.

The conditioning surfactants are cosmetically acceptable and suitable for topical application to the hair.

Suitable conditioning surfactants are selected from cationic surfactants, used singly or in admixture. Examples include quaternary ammonium hydroxides or salts thereof, e.g. chlorides.

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Suitable cationic surfactants for use in hair conditioners of the invention include cetyltrimethylammonium chloride, behenyltrimethylammonium chloride, cetylpyridinium chloride, tetramethylammonium chloride, tetraethylammonium chloride, octyltrimethylammonium chloride, dodecyltrimethylammonium chloride, hexadecyltrimethylammonium chloride, octyldimethylbenzylammonium chloride, decyldimethylbenzylammonium chloride, stearyldimethylbenzylammonium chloride, dioctadecyldimethylammonium chloride, dioctadecyldimethylammonium chloride, tallowtrimethylammonium chloride, cocotrimethylammonium chloride, and the corresponding hydroxides thereof. Further suitable cationic surfactants include those materials having the CTFA designations Quaternium-5, Quaternium-31 and Quaternium-18. Mixtures of any of the foregoing materials may also be suitable. A particularly useful cationic surfactant for use in hair conditioners of the invention is cetyltrimethylammonium chloride, available commercially, for example as DEHYQUART, ex Henkel.

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In conditioners of the invention, the level of cationic surfactant is preferably from 0.01 to 10%, more preferably 0.05 to 5%, most preferably 0.1 to 2% by weight of the composition.

The conditioning gel phase of the invention advantageously incorporates a fatty alcohol material. The combined use of fatty alcohol materials and cationic

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surfactants in conditioning compositions is believed to be especially advantageous, because this leads to the formation of a lamellar phase, in which the cationic surfactant is dispersed.

Representative fatty alcohols comprise from 8 to 22 carbon atoms, more preferably 16 to 20. Examples of suitable fatty alcohols include cetyl alcohol, stearyl alcohol and mixtures thereof. The use of these materials is also advantageous in that they contribute to the overall conditioning properties of compositions of the invention. The level of fatty alcohol material in conditioners of the invention is conveniently from 0.01 to 10%, preferably from 0.1 to 5% by weight of the composition. The weight ratio of cationic surfactant to fatty alcohol is suitably from 10:1 to 1:10, preferably from 4:1 to 1:8, optimally from 1:1 to 1:4.

According to the invention the conditioning gel phase is formed prior to incorporation of the PEC. Typically, this involves mixing fatty alcohol with the cationic surfactant in water at temperature and mixing. When cooled to around 35-45C the PEC is added. Preferably, the composition is homogenized prior to incorporation of the PEC to prevent breakage of the PEC particles. The composition is then further cooled to room temperature before further ingredients such as perfume, silicone, preservative and other benefit agents are added.

The resulting formulation comprising a conditioning gel phase may also comprises a silicone. The silicone may be added before or after the PEC however it is preferred that the PEC is added immediately after formation of the conditioning gel phase.

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The silicone is insoluble in the aqueous matrix of the composition and so is present in an emulsified form, with the silicone present as dispersed particles.

Suitable silicones include polydiorganosiloxanes, in particular polydimethylsiloxanes which have the CTFA designation dimethicone. Also suitable for use compositions of the invention (particularly shampoos and conditioners) are polydimethyl siloxanes having hydroxyl end groups, which have

the CTFA designation dimethiconol. Also suitable for use in compositions of the invention are silicone gums having a slight degree of cross-linking, as are described for example in WO 96/31188. These materials can impart body, volume and stylability to hair, as well as good wet and dry conditioning. The viscosity of the emulsified silicone itself (not the emulsion or the final hair conditioning composition) is typically at least 10,000 cst. In general we have found that conditioning performance increases with increased viscosity. Accordingly, the viscosity of the silicone itself is preferably at least 60,000 cst,

most preferably at least 500,000 cst, ideally at least 1,000,000 cst. Preferably the

viscosity does not exceed 10⁹ cst for ease of formulation.

Emulsified silicones for use in hair shampoos and conditioners of the invention will typically have an average silicone particle size in the composition of less than 30, preferably less than 20, more preferably less than 10 microns. We have found that reducing the particle size generally improves conditioning performance. Most preferably the average silicone particle size of the emulsified silicone in the composition is less than 2 microns, ideally it ranges from 0.01 to 1 micron. Silicone emulsions having an average silicone particle size of \leq 0.15 microns are generally termed microemulsions.

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Particle size may be measured by means of a laser light scattering technique, using a 2600D Particle Sizer from Malvern Instruments.

Suitable silicone emulsions for use in the invention are also commercially available in a pre-emulsified form.

Examples of suitable pre-formed emulsions include emulsions DC2-1766, DC2-1784, and microemulsions DC2-1865 and

30 DC2-1870, all available from Dow Corning. These are all emulsions/microemulsions of dimethiconol. Cross-linked silicone gums are also

available in a pre-emulsified form, which is advantageous for ease of formulation. A preferred example is the material available from Dow Corning as DC X2-1787, which is an emulsion of cross-linked dimethiconol gum. A further preferred example is the material available from Dow Corning as DC X2-1391, which is a microemulsion of cross-linked dimethiconol gum.

A further preferred class of silicones for inclusion in shampoos and conditioners of the invention are amino functional silicones. By "amino functional silicone" is meant a silicone containing at least one primary, secondary or tertiary amine group, or a quaternary ammonium group.

Examples of suitable amino functional silicones include:

(i) polysiloxanes having the CTFA designation "amodimethicone", and thegeneral formula:

$$HO-[Si(CH_3)_2-O-]_x-[Si(OH)(CH_2CH_2CH_2-NH-CH_2CH_2NH_2)-O-]_y-H$$

in which x and y are numbers depending on the molecular weight of the polymer, generally such that the molecular weight is between about 5,000 and 500,000.

(ii) polysiloxanes having the general formula:

$$R_aG_{3-a}$$
-Si(OSiG₂)_n-(OSiG_b R_{2-b})_m-O-SiG_{3-a}- R_a

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G is selected from H, phenyl, OH or C₁₋₈ alkyl, e.g. methyl;

a is 0 or an integer from 1 to 3, preferably 0;

b is 0 or 1, preferably 1;

m and n are numbers such that (m + n) can range from 1 to 2000,

30 preferably from 50 to 150;

m is a number from 1 to 2000, preferably from 1 to 10;

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n is a number from 0 to 1999, preferably from 49 to 149, and R is a monovalent radical of formula $-C_qH_{2q}L$ in which q is a number from 2 to 8 and L is an aminofunctional group selected from the following:

 $5 - N(R'')_2$

 $-N^{+}(R^{"})_{3}A^{-}$

 $-N^{+}H(R^{"})_{2}A^{-}$

 $-N^{+}H_{2}(R^{"})A^{-}$

-N(R")-CH₂-CH₂-N⁺H₂(R") A⁻

in which R is selected from H, phenyl, benzyl, or a saturated monovalent hydrocarbon radical, e.g. C₁₋₂₀ alkyl, and;

A is a halide ion, e.g. chloride or bromide.

- Suitable amino functional silicones corresponding to the above formula include those polysiloxanes termed "trimethylsilylamodimethicone" as depicted below, and which are sufficiently water insoluble so as to be useful in compositions of the invention:
- 20 Si(CH₃)₃ O [Si(CH₃)₂ O]_X [Si (CH₃) (R NH CH₂CH₂ NH₂) O]_y Si (CH₃)₃

wherein x + y is a number from about 50 to about 500, and wherein R is an alkylene group having from 2 to 5 carbon atoms. Preferably, the number x + y is in the range of from about 100 to about 300.

(ii) quaternary silicone polymers having the general formula:

$$\{(R^1)(R^2)(R^3) N^+ CH_2CH(OH)CH_2O(CH_2)_3[Si(R^4)(R^5)-O-]_n-Si(R^6)(R^7)-(CH_2)_3-O-CH_2CH(OH)CH_2N^+(R^8)(R^9)(R^{10})\}$$
 (X⁻)₂

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wherein R¹ and R¹⁰ may be the same or different and may be independently selected from H, saturated or unsaturated long or short chain alk(en)yl, branched chain alk(en)yl and C₅-C₈ cyclic ring systems;

R² thru' R⁹ may be the same or different and may be independently selected from H, straight or branched chain lower alk(en)yl, and C₅-C₈ cyclic ring systems;

n is a number within the range of about 60 to about 120, preferably about 80, and

X is preferably acetate, but may instead be for example halide, organic carboxylate, organic sulphonate or the like.

Suitable quaternary silicone polymers of this class are described in EP-A-0 530 974.

Amino functional silicones suitable for use in conditioners of the invention will typically have a mole % amine functionality in the range of from about 0.1 to about 8.0 mole %, preferably from about 0.1 to about 5.0 mole %, most preferably from about 0.1 to about 2.0 mole %. In general the amine concentration should not exceed about 8.0 mole % since we have found that too high an amine concentration can be detrimental to total silicone deposition and therefore conditioning performance.

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The viscosity of the amino functional silicone is not particularly critical and can suitably range from about 100 to about 500,000 cst.

Specific examples of amino functional silicones suitable for use in the invention are the aminosilicone oils DC2-8220, DC2-8166, DC2-8466, and DC2-8950-114 (all ex Dow Corning), and GE 1149-75, (ex General Electric Silicones).

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Also suitable are emulsions of amino functional silicone oils with non ionic and/or cationic surfactant.

Suitably such pre-formed emulsions will have an average amino functional silicone particle size in the shampoo composition of less than 30, preferably less than 20, more preferably less than 10 microns. Again, we have found that reducing the particle size generally improves conditioning performance. Most preferably the average amino functional silicone particle size in the composition is less than 2 microns, ideally it ranges from 0.01 to 1 micron. Silicone emulsions having an average silicone particle size of \leq 0.15 microns are generally termed microemulsions.

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Pre-formed emulsions of amino functional silicone are also available from suppliers of silicone oils such as Dow Corning and General Electric. Specific examples include DC929 Cationic Emulsion, DC939 Cationic Emulsion, and the non-ionic emulsions DC2-7224, DC2-8467, DC2-8177 and DC2-8154 (all ex Dow Corning).

An example of a quaternary silicone polymer useful in the present invention is the material K3474, ex Goldschmidt.

The total amount of silicone incorporated into compositions of the invention depends on the level of conditioning desired and the material used. A preferred amount is from 0.01 to about 10% by weight of the total composition although these limits are not absolute. The lower limit is determined by the minimum level to achieve conditioning and the upper limit by the maximum level to avoid making the hair and/or skin unacceptably greasy.

We have found that a total amount of silicone of from 0.3 to 5%, preferably 0.5 to 3%, by weight of the total composition is a suitable level.

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Polyelectrolyte complexes (PECs) are described in U.S. Patent Application Publication Nos. 2005/0089494 and 2006/0251603, both of which are incorporated herein by reference with regard to the nature of PECs and their methods for manufacture. As outlined in these publications, the formation of PECs is not simply due to the presence of oppositely charged polymers in a composition. A phase diagram may be produced to show the polymer-polymer species present when concentrations of the oppositely charged polymers are varied (thus varying the ratios of the polymers to each other). Based on the phase diagram, the ratio of polymers in terms of weight percent, moles, etc. at which PECs are formed can be determined. For PVMMA/polyquat-28 PECs, a preferred weight ratio is 1:9 PVMMA:polyquat-28. However, PECs may exist at other weight percent ratios. For example, PVMMA/polyquat-28 PECs can be formed around the 1:9 weight percent ratio, such that a ratio slightly higher or lower than 1:9 will have some amount of PECs present. The weight percent ratio can range from about 1:8 to about 1:10 PVMMA:polyquat-28.

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In addition to weight percent ratios, the formation of PECs may be determined based on the charge-charge ratio of polymers. PECs are usually formed when the charge-charge ratio of the polymers is 1:1. Therefore, PECs are likely to form when, *e.g.*, each negative charge of a polymer is balanced by a positive charge of another polymer. However, PECs may exist at other charge-charge ratios. For example, PECs can be formed around the 1:1 charge-charge ratio, such that a ratio slightly higher or lower than 1:1 will have at least some amount of PECs present. A phase diagram can be produced to determine the presence of PECs at varying charge-charge ratios.

Contemplated herein are PECs formed between polymers comprising cationic charges and polymers comprising anionic charges. However, phase diagrams may be constructed to determine the suitability of PEC formation for any polymer-polymer system, so long as the polymers interact through ionic bonds. The compositions of the present invention may comprise a polyelectrolyte complex

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wherein a cationic polymer comprises one or more monomeric units with one or more quaternary ammonium nitrogen moieties, for example, vinylpyrrolidone/methacrylamidopropyl trimethylammonium chloride copolymer (polyquaternium-28). Non-limiting examples of other cationic polymers that may be used in forming PECs include other polyquat polymers such as polyquaternium-7, polyquaternium-10, or polyquaternium-11. The compositions of the present invention may comprise a polyelectrolyte complex wherein an anionic polymer comprises monomeric units having ionizable carboxylic acid moieties, for example, a methylvinylether/maleic acid copolymer. Another example of an anionic polymer that may be used in forming PECs is vinylpyrrolidone/acrylates/lauryl methacrylate copolymer.

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Polymers with ionizable groups, such as carboxylic acid groups or tertiary amine groups, may require the adjustment of the pH in order for a polyelectrolyte complex to form. Adjustment of the pH alters the percent of ionizable groups that are ionized and thus adjusts the number of groups that may interact through ionic bonds. Production of phase diagrams at varying pHs can be used to determine the proper pH for PEC formation. For PVMMA/polyguat-28 PECs, a pH range of from about 6.5 to about 7.5 may be used for PEC formation. The pH of a composition may be adjusted using neutralizing/buffering agents. For acidic groups such as carboxylic acid, a basic neutralizer can be used, such as a hydroxide compound. Suitable hydroxide compounds include strong bases such as sodium hydroxide. For basic groups, an acidic neutralizer can be used, such as citric acid. Any suitable concentration of neutralizer can be used, including from about 0.1 wt. % to about 30 wt. preferably from about 5 wt. % to about 20 wt. %. So long as the PECs have formed under desired pH conditions, the final pH of any resulting composition comprising the PECs may be the same or different from the pH at which the PECs were formed, so long as the PECs remain intact.

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The PVMMA/polyquat-28 PECs have the ability to mend split-ends of hair fibers. Hair has a net negative charge. Without being bound to any particular theory, PECs are believed to be substantive to the hair fiber and adhere the ends of a split-end together through adhesive properties. The positive charges of the polyquat-28 polymer are attracted to the negatively charged hair fibers and are substantive to the hair. Since the negatively charged PVMMA polymer is complexed to the polyquat-28, the PVMMA also is drawn to the hair. The ends of the split-end are then adhered together through the adhesive action of multiple PECs interacting with the ends of the split-end and/or each other (*e.g.*, in filmformation where the film contracts while drying). Additionally, the surface tension experienced by the ends of the split-end due to the interaction of water during filmformation assists in adhering the ends together.

To the extent that the PECs exist in the form of suspended or emulsified particles, the particle size of PECs should allow the PECs access to the ends of a spilt-end in order for the PECs to mend damaged hair fibers. As long as the PECs have access to the ends of a split-end, the particle size distribution can have any suitable average PEC particle size. Suitable average PEC particle sizes can include, e.g., an average PEC particle size of from about 0.5 micrometer to about 40 micrometers. In some embodiments, the compositions of the present invention have a PEC particle distribution with an average PEC particle size of about 5 ± 3 micrometers. The compositions of the present invention also can include PEC particles that exist within an interlocking microgel structure.

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The compositions of the present invention can incorporate PECs at higher concentrations than previously recognized as achievable in the art. For instance, the compositions of the present invention can include PECs of PVMMA/polyquat-28 at a concentration of from 0.1 to 8 wt. %. The concentration of PECs in a hair care composition may be varied. The compositions of the present invention may contain the polyelectrolyte complex in a concentration of from about 0.1% to about

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4% based on the total weight of the composition. Preferably, the compositions of the present invention contain the polyelectrolyte complex is in a concentration of from about 0.5% to about 1.5% based on the total weight of the composition.

Compositions can be meant for rinse-out, leave-in, and/or overnight treatments. The hair care compositions of the present invention may comprise mixing a polyelectrolyte complex with one or more components selected from aqueous carriers, surfactants, neutralizers, fragrances, conditioning agents, emulsifiers, thickening agents, preservatives, gelling agents, silicones, hair fixatives, humectants and moisturizers to form a hair care composition.

An embodiment of a composition of the present invention can include, for example, 1-2 wt. % PEC of PVMMA copolymer and polyquaternium-28 in a 1:9 ratio.

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The compositions of the present invention may be used in any suitable method for treating hair. For example, compositions described herein may be used for conditioning a hair fiber by applying a composition to the hair fiber and rinsing the hair fiber with water. After rinsing, the hair fiber may be dried with a blow dryer, combed, and/or styled using styling products or compositions. Additionally, compositions described herein may be used to repair a split-end of a hair fiber by applying a composition to the split-end and rinsing the split-end with water. This method also may be followed by blow drying, combing, and/or styling. The compositions described herein further may be used in a method of thickening a hair fiber by applying to the hair fiber a composition of the present invention and rinsing the hair with water.

Some embodiments of the compositions of the present invention include compositions with the formulations of PVMMA and polyquat-28 as in Table 1.

Table 1

wt. % active PECs:	1%	2%	4%	6%	8%
Water	7.775	15.55	31.1	46.65	62.2
PVM/MA Copolymer	0.1	0.2	0.4	0.6	0.8
Sodium Hydroxide, Water (20%					
active)	0.125	0.25	0.5	0.75	1
Polyquaternium-28, Water (20%					
active)	4.5	9	18	27	36
Remainder of composition	87.5	75	50	25	0
Total	100	100	100	100	100

In a second aspect there is provided a method according to claim 14.

5 In a third aspect there is provided a method according to claim 15.

EXAMPLE 1

The following is a hair conditioning composition comprising PEC and lamellar gel phase. The PEC is represented by PQ-28 and PVM/MA copolymer.

Ingredients	Wt. %	
Lactic Acid	0.335	
Stearamidopropyl dimethylamine	1.25	
Behenyl trimmonium chloride	0.875	
Cetostearyl alcohol	5.000	
EDTA	0.1	
Aminosilicone	0.75	
Preservative	0.4	
Perfume	0.6	
Sodium Chloride	0.1	
PVM/MA copolymer	0.1	
Sodium Hydroxide	0.025	
PQ-28	0.9	
Water	To 100	

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The formulation is made by a process according to Example 2.

EXAMPLE 2

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- 1. Heat water and lactic acid to 82-85 ℃
- 2. Add stearamidopropyl dimethylamine, behenyl trimmonium chloride and cetostearyl alcohol. Homogenise with stirrer at ~130rpm.
- 3. Begin to cool. Once batch reaches 65 °C, add quench water slowly down side of vessel, ensuring stirrer speed has been reduced (~80rpm).
- 4. At ~50 °C, add EDTA (pre-dissolved in hot water)
- 5. Once temperature is 40-45 °C, add PEC premix. Leave to stir for 10 minutes.
- 6. Below 37°C, add silicone, fragrance, emotives and preservatives.
- 15 7. Back weigh any water lost during processing.

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CLAIMS

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- 1. Conditioning composition obtainable by making a conditioning gel phase and then adding polyelectrolyte complex (PEC) to the gel phase, wherein the conditioning gel phase comprises a cationic surfactant and/or a fatty alcohol and wherein the composition comprises from 0 to 2% by weight anionic surfactant.
- A conditioning composition according to claim 1 wherein the PEC
 comprises a cationic polymer and an anionic polymer.
 - 3. The composition of claim 1 or 2 wherein the PEC is in an amount of from 0.1% to 8% based on the total weight of the composition.
- 4. The composition of any preceding claim wherein the PEC is in an amount of from 0.5% to 1.5% based on the total weight of the composition.
 - 5. The composition of any of claims 2 to 4 wherein the cationic polymer comprises one or more monomeric units with one or more quaternary ammonium nitrogen moieties.
 - 6. The composition of any of claims 2 to 5 wherein the cationic polymer is polyquaternium-28.
- 7. The composition of any claims 2 to 6 wherein the anionic polymer comprises monomeric units having ionizable carboxylic acid moieties.
 - 8. The composition of any of claims 2 to 7 wherein the anionic polymer is a methylvinylether/maleic acid copolymer.

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- 9. The composition of any preceding claim wherein the PEC is in the form of a microgel.
- 10. The composition of any preceding claim wherein the PEC has an average particle size of 5 μm.

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- 11. The composition of any of claims 2 to 10 wherein the cationic polymer and anionic polymer are in the weight ratios of 1:8 to 1:10 based on weight of active.
- 12. The composition of any preceding claim wherein the fatty alcohol has from 12 to 20 carbons, more preferably from 16 to 18 carbons.
- 13. The composition of any preceding claim wherein the conditioning gel phasecomprises behenyl trimmonium chloride.
 - 14. A method conditioning a hair fiber by applying to the hair a composition according to any preceding claim and massaging hair for from 5 seconds to 10 minutes and then rinsing with water.
 - 15. A method of repairing split ends of a hair fiber by applying to the hair a composition according to any preceding claim and massaging hair for from 5 seconds to 10 minutes and then rinsing with water.