## (19) World Intellectual Property Organization

International Bureau



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## (10) International Publication Number WO 2012/054029 A1

### (43) International Publication Date 26 April 2012 (26.04.2012)

- (51) International Patent Classification: A61K 8/72 (2006.01) A61K 9/14 (2006.01)
- (21) International Application Number:

PCT/US2010/053360

(22) International Filing Date:

20 October 2010 (20.10.2010)

(25) Filing Language:

**English** 

(26) Publication Language:

English

(30) Priority Data:

61/394,966 20 October 2010 (20.10.2010)

US

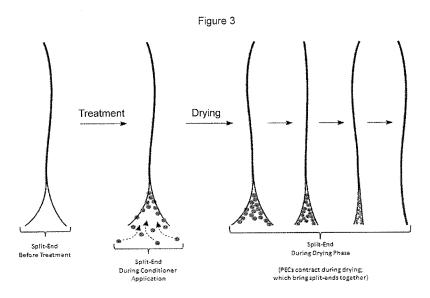
- (71) Applicant (for all designated States except US): AL-BERTO-CULVER COMPANY [US/US]; 2525 Armitage Avenue, Melrose Park, Illinois 60160 (US).
- (72) Inventors; and
- Inventors/Applicants (for US only): WRIGHT, Michael [US/US]; 318 Weatherford Lane, Naperville, Illinois 60565 (US). SZERSZEN, Margaret [US/US]; 7925 W. Tiffany Ct., Palos Hills, Illinois 60465 (US). COHEN, Jason [US/US]; W2801 Oakridge Drive, Appleton, Wisconsin 54915 (US). PETROSKI, Dan [US/US]; 1638 Orchard Avenue, Schaumburg, Illinois 60193 (US). EA-GAN, Deborah [US/US]; 1040 Rice, Bellwood, Illinois 60104 (US). FELSKI, Chris [US/US]; 618 W. Briar Pl., Unit 1, Chicago, Illinois 60657 (US).

- (74) Agents: SPINA, Kenneth P. et al.; Leydig, Voit & Mayer, Ltd., Two Prudential Plaza, Ste. 4900, 180 North Stetson Avenue, Chicago, Illinois 60601-6731 (US).
- (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, 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, KM, 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, PE, PG, PH, PL, PT, RO, RS, RU, 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, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, 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, ML, MR, NE, SN, TD, TG).

#### Published:

with international search report (Art. 21(3))

#### (54) Title: HAIR-MENDING COMPOSITIONS AND ASSOCIATED METHODS



(57) Abstract: Disclosed herein are hair-mending compositions containing a polyelectrolyte complex. Also disclosed are methods of their use, methods of their manufacture, methods of testing their efficacy, and media methods involving hair mending.



#### HAIR-MENDING COMPOSITIONS AND ASSOCIATED METHODS

#### BACKGROUND OF THE INVENTION

[0001] Hair care compositions are often formulated to have multiple properties. For example, a hair conditioner may be formulated to condition hair and also may be formulated to repair hair. Hair care compositions with multiple properties are attractive to a consumer in that fewer compositions need be applied to the hair to achieve a desired effect. However, to formulate such a hair care composition, multiple ingredients are often necessary. As the number of ingredients increases, the complexity of the composition increases. The long term stability of such a composition can become compromised due to incompatibilities among the many ingredients.

[0002] Furthermore, due to the large number of ingredients in many hair care compositions, formulators are not only faced with the challenges of finding the proper ingredients to provide the desired cosmetic properties but are also faced with the challenge of manufacturing these complicated compositions. Such challenges include developing production protocols to produce a composition both on a small scale for initial testing and on a large scale for commercial marketing. A greater number of ingredients may translate into a greater number of manufacturing containers required, which itself translates into more time and expense to produce the composition. Cosmetic compositions thus are complicated mixtures of ingredients, and with such complexity, require careful consideration when developing production protocols.

[0003] Hair care composition ingredients include conditioning agents, which are often positively charged compounds. Hair fibers have a net negative charge. The positive charge of the conditioning agents is attracted to the overall negative charge of the hair fibers, making the positively charged conditioning agents substantive to the hair. This substantive nature allows the conditioning agent to interact with the hair in order for the conditioning agent to provide its conditioning effect. The conditioning effect can include restoring the hair fiber after damage to the hair, such as repairing split-ends.

[0004] Conditioning agents known in the art include quaternary ammonium nitrogen compounds, known as "quats," and polymers comprising moieties of such compounds, known as "polyquats." The polyquats include polyquaternium-28, a vinylpyrrolidone/methacrylamidopropyl trimethylammonium chloride copolymer. It has been found that the combination of polyquat-28 and methylvinylether/maleic acid copolymer

(PVMMA) at particular ratios produces a polyelectrolyte complex (PEC) that repairs splitend damage (U.S. Patent Application Publication Nos. 2005/0089494 and 2006/0251603, both assigned to ISP Investments Inc.). However, within the context of complicated hair care formulations, 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.

[0005] Also, although PVMMA:polyquat-28 PECs consist of only two component ingredients, the method of producing these PECs is described in the ISP Investments applications as comprising two or three separate containers. Such a method is cumbersome, especially in view of any additional containers required to incorporate additional ingredients. The dedication of two or three containers in a large-scale production facility for the making of one complex is inefficient and costly.

[0006] Additionally, when a consumer purchases hair care products, the consumer does so based on the properties of the hair care product. For example, a consumer with frizzy hair may purchase a product to de-frizz the hair, and a consumer with split-ends may purchase a product to repair the split-ends. Thus, consumers may purchase hair care products having properties desirable for a particular hair type or hair problem. In order for consumers to compare hair care product compositions to make a decision regarding which to purchase, the beneficial properties of the compositions should be clearly communicated to the consumers. One method is to use still photography, such as described in the ISP Investments applications. However, additional methods are needed.

[0007] It would therefore be desirable to formulate a stable hair care composition that includes intact PECs in combination with other hair benefit agents, especially charged hair benefit agents. Also, there is a need for a more efficient process for making PECs and hair care compositions containing PECs. Further, there is a need for the development of test methods that can show improved results of split-end mending of a hair care composition such that the benefits of the hair care composition can then be effectively communicated to consumers. There also is a need for the development of additional methods that can show the benefits of a hair care composition such that the benefits can then be effectively

communicated to consumers. The present invention provides these advantages. Additional advantages of the invention, as well as additional inventive features, will be apparent from the description of the invention provided herein.

#### BRIEF SUMMARY OF THE INVENTION

[0008] In one aspect, the present invention provides a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer in the form of an emulsion with improved freeze-thaw stability relative to comparative compositions containing such polyelectrolyte complexes.

[0009] In another aspect, the present invention provides a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, wherein the viscosity of the composition is greater than 3000 cps.

[0010] In another aspect, the present invention provides a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer; from about 1% to about 5% of a thickening agent; about 10% or greater of propylene glycol; and about 5% or greater of an emollient; wherein the percentages are based on the total weight of the composition.

**[0011]** In yet another aspect, the present invention provides a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer; from about 3% to about 5% of a thickening agent; and about 1% or less of an emollient; wherein the percentages are based on the total weight of the composition.

[0012] 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.

[0013] Furthermore, the present invention provides a method for improving the freeze-thaw stability of a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, the method comprising adding to the hair care composition from about 1% to about 5% of a thickening agent, wherein the percentage is based on the total weight of the composition.

[0014] Additionally, the present invention provides a method for improving the freeze-thaw stability of a hair care composition comprising a polyelectrolyte complex, the method comprising adding to the hair care composition from about 10% or greater of propylene glycol, wherein the percentage is based on the total weight of the composition.

[0015] Furthermore, an aspect of the present invention provides a method of producing a polyelectrolyte complex, the method comprising hydrating a methylvinylether/maleic acid copolymer having repeating units of the formula (Formula I)

in a container; reacting the methylvinylether/maleic acid copolymer with a base such as, *e.g.*, sodium hydroxide, in the container in an amount sufficient to at least partially neutralize the acidic groups thereof, to form an intermediate mixture; heating the intermediate mixture in the container; adding to the intermediate mixture in the container a cationic copolymer of a vinylpyrrolidone of the formula (Formula II)

and a methacrylamidopropyl trimethylammonium chloride of the formula (Formula III)

mixing the polymers in the container at high shear force and at a temperature of at least about 50-60 °C to produce the polyelectrolyte complex.

[0016] The present invention also provides a method of producing a polyelectrolyte complex, the method comprising hydrating a methylvinylether/maleic acid copolymer having repeating units of the formula I in a container; reacting the methylvinylether/maleic acid copolymer with a 20% sodium hydroxide solution in the container, wherein the sodium hydroxide is added in an amount sufficient to achieve a pH of from about 6.9 to about 7.0, to form an intermediate mixture; heating the intermediate mixture in the container to a

temperature of from about 50 °C to about 60 °C; adding to the intermediate mixture in the container a cationic copolymer of a vinylpyrrolidone of the formula II and a methacrylamidopropyl trimethylammonium chloride of the formula III; and mixing the polymers in the container for at least about 10 minutes at high shear force and at a temperature of at least about 50-60 °C to produce the polyelectrolyte complex.

[0017] The present invention also provides a method of producing a hair care composition comprising producing a polyelectrolyte complex, comprising the steps of hydrating a methylvinylether/maleic acid copolymer having repeating units of the formula I in a container; reacting the methylvinylether/maleic acid copolymer with a base such as, *e.g.*, sodium hydroxide, in the container in an amount sufficient to at least partially neutralize the acidic groups thereof, to form an intermediate mixture; heating the intermediate mixture in the container; adding to the intermediate mixture in the container a cationic copolymer of a vinylpyrrolidone of the formula II and a methacrylamidopropyl trimethylammonium chloride of the formula III; mixing the polymers in the container at high shear force and at a temperature of at least about 50-60 °C to produce the polyelectrolyte complex; adding to the polyelectrolyte complex in the container one or more additional components selected from aqueous carriers, surfactants, fragrances, conditioning agents, emollients, emulsifiers, thickening agents, preservatives, gelling agents, silicones, hair fixatives, humectants and moisturizers; and mixing the resulting composition in the container.

[0018] The present invention also provides a method of producing a hair care composition comprising producing a polyelectrolyte complex between a methylvinylether/maleic acid copolymer and a cationic copolymer of a vinylpyrrolidone and a methacrylamidopropyl trimethylammonium chloride, comprising the steps of hydrating a methylvinylether/maleic acid copolymer having repeating units of the formula I in a first container; reacting the methylvinylether/maleic acid copolymer with a base such as, *e.g.*, sodium hydroxide, in the first container in an amount sufficient to at least partially neutralize the acidic groups thereof, to form an intermediate mixture; heating the intermediate mixture in the first container; adding to the intermediate mixture in the first container a cationic copolymer of a vinylpyrrolidone of the formula II and a methacrylamidopropyl trimethylammonium chloride of the formula III; mixing the polymers in the first container at high shear force and at a temperature of at least about 50-60 °C to produce the polyelectrolyte complex; mixing in a second container one or more components selected from aqueous carriers, surfactants, fragrances, conditioning agents, emollients, emulsifiers, thickening

agents, preservatives, gelling agents, silicones, hair fixatives, humectants and moisturizers; and mixing together in the first or second container the polyelectrolyte complex of the first container and the mixed components of the second container.

[0019] Furthermore, the present invention provides a method of graphically reconstructing a repair of a split-end of a hair fiber, the method comprising securing a hair fiber to a surface, wherein the hair fiber secured to the surface has a split-end or is damaged while on the surface to produce a split-end; associating the surface with a magnifying device, wherein the split-end is positioned within the field of view of the magnifying device; associating an image capture device with the magnifying device so as to capture an enlarged image of the field of view of the magnifying device; treating the split-end with a composition, wherein the composition repairs the split-end and wherein the split-end fuses together; and via the image capture device associated with the magnifying device, capturing an image of the repair of the split-end during treatment with the composition, and storing a representation of the captured image of the repair for subsequent retrieval.

[0020] The present invention also provides a method of quantitatively assessing the degree of split-end hair fiber repair, the method comprising obtaining a tress of hair, wherein the hair is twice-bleached hair; combing and blow drying the tress of hair to produce at least one hair fiber of the tress having a split-end; removing at least one hair fiber having a split-end from the tress; optionally marking the at least one removed hair fiber; capturing a first image of the split-end of the at least one removed hair fiber and storing an electronic representation of the image; associating the at least one marked hair fiber with the tress from which the hair fiber was removed; treating the tress and the at least one marked hair fiber with a composition; removing the at least one marked hair fiber from the tress; capturing a second image of the end of the at least one marked hair fiber, wherein the end is the same end captured for the first image of the split-end of the at least one removed hair fiber; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair; and summing the assigned numbers for all marked hair fibers and dividing the sum by the total number of marked hair fibers.

[0021] One embodiment of the present invention provides a method of quantitatively assessing the degree of split-end hair fiber repair, the method comprising obtaining a tress of hair; producing at least one hair fiber of the tress to have a spilt-end; removing at least one hair fiber having a split-end from the tress; marking the at least one removed hair fiber; capturing a first image of the split-end of the at least one removed hair fiber and storing an

electronic representation of the image; associating the at least one marked hair fiber with the tress from which the hair fiber was removed; treating the tress and the at least one marked hair fiber with a composition, wherein the composition comprises a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, from about 1% to about 5% of a thickening agent, about 10% or greater of propylene glycol, and about 5% or greater of an emollient, wherein the percentages are based on the total weight of the composition; removing the at least one marked hair fiber from the tress; capturing a second image of the end of the at least one marked hair fiber, wherein the end is the same end captured for the first image of the split-end of the at least one removed hair fiber; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair; and summing the assigned numbers for all marked hair fibers and dividing the sum by the total number of marked hair fibers.

Another embodiment of the present invention provides a method of quantitatively [0022] assessing the degree of split-end hair fiber repair, the method comprising obtaining a tress of hair; producing at least one hair fiber of the tress to have a spilt-end; removing at least one hair fiber having a split-end from the tress; marking the at least one removed hair fiber; capturing a first image of the split-end of the at least one removed hair fiber and storing an electronic representation of the image; associating the at least one marked hair fiber with the tress from which the hair fiber was removed; treating the tress and the at least one marked hair fiber with a composition, wherein the composition comprises a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, from about 3% to about 5% of a thickening agent, and about 1% or less of an emollient, wherein the percentages are based on the total weight of the composition; removing the at least one marked hair fiber from the tress; capturing a second image of the end of the at least one marked hair fiber, wherein the end is the same end captured for the first image of the splitend of the at least one removed hair fiber; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair; and summing the assigned numbers for all marked hair fibers and dividing the sum by the total number of marked hair fibers.

[0023] Another embodiment of the present invention provides a method of quantitatively assessing the degree of split-end hair fiber repair, the method comprising obtaining a tress of hair; producing at least one hair fiber of the tress to have a spilt-end; removing at least one hair fiber having a split-end from the tress; marking the at least one removed hair fiber; capturing a first image of the split-end of the at least one removed hair fiber and storing an

electronic representation of the image; associating the at least one marked hair fiber with the tress from which the hair fiber was removed; treating the tress and the at least one marked hair fiber with a composition, wherein the composition comprises a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, wherein the viscosity of the composition is greater than 3000 cps; removing the at least one marked hair fiber from the tress; capturing a second image of the end of the at least one marked hair fiber, wherein the end is the same end captured for the first image of the split-end of the at least one removed hair fiber; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair; and summing the assigned numbers for all marked hair fibers and dividing the sum by the total number of marked hair fibers.

Another embodiment of the present invention provides a method of quantitatively [0024] assessing the degree of split-end hair fiber repair, the method comprising obtaining a tress of hair; producing at least one hair fiber of the tress to have a spilt-end; removing at least one hair fiber having a split-end from the tress; marking the at least one removed hair fiber; capturing a first image of the split-end of the at least one removed hair fiber and storing an electronic representation of the image; associating the at least one marked hair fiber with the tress from which the hair fiber was removed; treating the tress and the at least one marked hair fiber with a composition, wherein the composition comprises a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer in the form of an emulsion with improved freeze-thaw stability relative to comparative compositions containing such polyelectrolyte complexes; removing the at least one marked hair fiber from the tress; capturing a second image of the end of the at least one marked hair fiber, wherein the end is the same end captured for the first image of the split-end of the at least one removed hair fiber; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair; and summing the assigned numbers for all marked hair fibers and dividing the sum by the total number of marked hair fibers.

#### BRIEF DESCRIPTION OF THE DRAWINGS

[0025] Figure 1 is a bar graph that shows percent mending determined using a method of the present invention with rinse-off conditioners in three cycles. Numbers in parentheses are percent polyelectrolyte complex (PEC). The number of fibers tested for the various compositions were 20, 100, and/or 50.

[0026] Figure 2 is a bar graph that shows percent mending determined using a method of the present invention with leave-in compositions in one treatment. Twenty fibers were tested for each composition.

[0027] Figure 3 is a drawing showing the mending of a split-end using polyelectrolyte complexes (PECs). The drawing shows a possible mechanism of split-end repair by PECs: PECs attach to the site of the split-end, form a network with themselves and the fiber assembly, and shrink as they dry binding the split end from within.

[0028] Figures 4A-4D show still photography of the mending of a variety of split-end types.

[0029] Figure 5 shows still photography taken from video of the mending of split-ends during the process of mending.

[0030] Figure 6 is a schematic diagram of an image data capture system in accordance with an embodiment of the invention.

#### DETAILED DESCRIPTION OF THE INVENTION

[0031] The present invention is predicated on the surprising and unexpected discovery of stable compositions containing polyelectrolyte complexes (PECs) and other hair benefit agents, particularly charged species, wherein the PECs remain intact and exhibit hair repair efficacy. It was unexpectedly found that the PECs can be combined with other charged hair benefit agents, such as, e.g., cationic conditioning agents, without disrupting the complex that defines the PEC structure. Due to this unexpected discovery, the present invention provides compositions that not only provide the hair mending effect of PECs but also provides the advantages of other hair benefit agents, such as, e.g., charged hair benefit agents, e.g., quaternary ammonium salts, cationic conditioning polymers, and the like. Suitable hair benefit agents that can be combined with PECs in accordance with the present invention include, for example, stearamidopropyl dimethylamine, polyquaternium-37, polyquaternium-7, polyquaternium-39, DC 5-7113 (silicone quaternium-16 and undeceth-11 and butyloctanol and undeceth-5), Abil<sup>®</sup> T Quat 60 (silicone quaternium-22), quaternium-80, Merquat<sup>®</sup> 2001 (polyquaternium-47), and acrylic acid/VP crosspolymer. Other suitable charged hair benefit agents are described herein.

[0032] The present invention also provides a one-container process for producing a PEC. The process of the present invention also allows for the production of PECs of the same particle size distribution as those prepared using conventional processes requiring two or

more separate containers. Thus, the present invention provides a more efficient and cost effective process for manufacturing PECs and hair care compositions containing PECs. The present invention also allows for concentrations of PECs greater than 4 wt. %, *e.g.*, 8 wt. %.

[0033] Additionally, the present invention has the advantage of providing methods that can graphically demonstrate the benefits of a hair care composition such that the benefits can then be effectively communicated to consumers. Consumers purchase hair care products based on the properties of the hair care products. Effectively communicating to these consumers the beneficial properties of a hair care product composition, then, is very important so that the consumers can make informed purchasing decisions when comparing competing hair care products.

The present invention further has the advantage of providing a test method that [0034] allows for showing improved results of split-end mending of a hair care composition such that the benefits of the hair care composition can then be effectively communicated to consumers. Most split-end mending test methods use virgin hair where the split-ends are mechanically induced. It is likely that virgin hair is used in order for the ingredients of a split-end mending composition to act solely on the mechanically induced damage. In other words, in virgin hair, there is no other damage to the hair besides the split-ends. Therefore, the mending composition directs its mending capabilities only to the split-end damage. The mending ingredients of such a composition, then, would not be wasted on other parts of the hair. However, it has been unexpectedly found that the use of hair with additional damage, e.g., due to bleaching, can be used instead of virgin hair in a split-end mending test method. This is counterintuitive since it would be expected that any mending composition would have its mending ingredients attracted to other damage of the hair and not just the split-ends, reducing the amount of split-end repair. It has been found by Applicants that the more damaged the hair, the better the mending of split-ends. Further, it has been found that, using more damaged hair, a high mending percentage can be achieved in a rinse-out product, e.g., greater than 50%, a level achievable with leave-in products.

[0035] In one aspect, the present invention provides a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer in the form of an emulsion with improved freeze-thaw stability relative to comparative compositions, *e.g.*, made by formulating the PECs described in U.S. Patent Application Publication Nos. 2005/0089494 and 2006/0251603 in conventional hair care base formulations.

[0036] The compositions of the present invention provide unexpected freeze-thaw

stability. Freeze-thaw stability is an important property of hair care compositions particularly for purposes of transport and storage which often exposes the compositions to low temperature extremes. Freeze-thaw stability is also important for products which remain on the hair and are thus exposed to temperature extremes outdoors. A composition containing PECs is not inherently unstable under freeze/thaw conditions. However, when combined with one or more fatty alcohols (*e.g.*, cetyl, stearyl, etc.), which have been used in the art to thicken conditioner compositions, at *e.g.*, about 3 wt. %, the formulation will be unstable during freeze/thaw. The composition will get grainy and chunky.

[0037] In addition to freeze/thaw instability, conventional compositions with PECs that contain about 5% fatty alcohol do not have long term high temperature stability. The viscosity of such a composition continues to increase over time (*e.g.*, over 100,000 cps after 2-3 months at 45 °C). The present inventors have found that the addition of Salcare<sup>®</sup> improves viscosity control; that is, viscosity is controlled to stay within, *e.g.*, about 20,000-40,000 cps.

[0038] The compositions of the present invention can contain a thickening agent. Thickening agents add viscosity to a composition such that a composition with a greater amount of a given thickening agent has a higher viscosity. The thickening agent can be, for example a quaternary ammonium nitrogen compound or polymer, for example polyquaternium-37, and for example where polyquaternium-37 is in a mixture with propylene glycol dicaprylate/dicaprate and polypropylene glycol-1 trideceth-6. Salcare® SC 96 from Ciba®/BASF, Basel, Switzerland is an example of such a mixture. Salcare® SC 96 is 65% polyquaternium-37, 25% propylene glycol dicaprylate/dicaprate, 10% PPG-1 trideceth-6. Other suitable thickening agents also can be added to the compositions of the present invention including, for example, xanthan gum, acrylic acid/VP crosspolymer (*e.g.*, Ultrathix<sup>TM</sup> P-100; ISP, Wayne, NJ, USA), PVP, PVP K-90, PEG-90M, and styrene/VP copolymer.

[0039] In another aspect, the present invention provides a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, wherein the viscosity of the composition is greater than 3000 cps.

[0040] The viscosity of the compositions of the present invention can be varied based on the amount of thickening agent and/or other ingredients added. The compositions of the present invention may comprise thickening agents in a concentration of from about 1 wt. % to about 10 wt. %. The thickening agent may be present in an amount in wt. %, e.g., from about

1% to about 10%, from about 1% to about 9%, from about 1% to about 8%, from about 1% to about 7%, from about 1% to about 5%, from about 1% to about 4%, from about 1% to about 3%, from about 1% to about 2%, from about 2% to about 10%, from about 2% to about 9%, 2% to about 8%, from about 2% to about 7%, from about 2% to about 3%, from about 2% to about 4%, from about 2% to about 3%, from about 3% to about 10%, from about 3% to about 3% to about 3% to about 3% to about 5%, from about 3% to about 4%, from about 4% to about 6%, from about 4% to about 9%, from about 4% to about 4% to about 5%, from about 4% to about 5%, from about 4% to about 5%, from about 5% to about 6% to about 6% to about 5% to about 5% to about 5% to about 6% to about 6% to about 6% to about 5% to about 5% to about 6% to ab

The viscosity of the compositions of the present invention can range, e.g., from [0041] about 3000 cps to about 100,000 cps, or, e.g., from about 20,000 cps to about 40,000 cps, as measured by RV T-B, 10 rpm, 1 min, at 25 °C for viscosities under 50,000 cps or RV T-C, 5 rpm, 1 min, at 25 °C for viscosities above 50,000 cps, but below 100,000 cps. The viscosity of the compositions of the present invention can range from about 3000 to about 100,000 cps, from about 4000 to about 100,000 cps, from about 5000 to about 100,000 cps, from about 6000 to about 100,000 cps, from about 7000 to about 100,000 cps, from about 8000 to about 100,000 cps, from about 9000 to about 100,000 cps, from about 10,000 to about 100,000 cps, from about 20,000 to about 100,000 cps, from about 30,000 to about 100,000 cps, from about 40,000 to about 100,000 cps, from about 50,000 to about 100,000 cps, from about 60,000 to about 100,000 cps, from about 70,000 to about 100,000 cps, from about 80,000 to about 100,000 cps, from about 90,000 to about 100,000 cps, from about 3000 to about 90,000 cps, from about 4000 to about 90,000 cps, from about 5000 to about 90,000 cps, from about 6000 to about 90,000 cps, from about 7000 to about 90,000 cps, from about 8000 to about 90,000 cps, from about 9000 to about 90,000 cps, from about 10,000 to about 90,000 cps, from about 20,000 to about 90,000 cps, from about 30,000 to about 90,000 cps, from about 40,000 to about 90,000 cps, from about 50,000 to about 90,000 cps, from about 60,000 to about 90,000 cps, from about 70,000 to about 90,000 cps, from about 80,000 to about 90,000 cps, from about 3000 to about 80,000 cps, from about 4000 to about 80,000 cps, from about 5000 to

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[0042] In one aspect of the invention, the composition contains a thickening agent, propylene glycol, and a relatively high concentration of an emollient, e.g., in an amount of 5 wt. % or greater. Suitable emollients can include, e.g., one or more fatty alcohols. Consumers may find hair care products containing 10 wt. % or more of propylene glycol to be unacceptable in that they may think such products would be heavy or sticky feeling, may notice hair taking too long to dry (due to the humectant properties of glycols), or may notice hair is hard to comb or not shiny. Surprisingly, however, it was found that including propylene glycol at 10% or greater in accordance with the present invention not only provided acceptable freeze-thaw stability but also may provide a composition that is more likely to be found acceptable by consumers. Additionally, Applicants have found that an emulsion in such compositions can accept a high level of glycol and not be affected (e.g., viscosity change, etc.). Applicants found that other polyhydric alcohols such as sorbitol and glycerin at 5 wt. %, did not provide for acceptable freeze-thaw stability. In some embodiments, the compositions of the present invention may contain propylene glycol in concentrations of about 10 wt. % or greater, about 20 wt. % or greater, about 30 wt. % or greater, about 40 wt. % or greater, or about 50 wt. % or greater.

[0043] The present invention also provides a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer; from about 1% to about 5% of a thickening agent; about 10% or greater of propylene glycol; and about 5% or greater of an emollient; wherein the percentages are based on the total weight of the composition.

[0044] In another aspect, the present invention provides a hair care composition that

includes a polyelectrolyte complex of a cationic polymer and an anionic polymer, in combination with from about 3% to about 5% of a thickening agent, and about 1% or less of an emollient, wherein the percentages are based on the total weight of the composition. In this aspect, the composition of the present invention is preferably substantially free of propylene glycol. As used herein, "substantially free of propylene glycol" means that the composition of the present invention contains less than about 1 wt. % propylene glycol, less than about 0.5 wt. % propylene glycol, less than about 0.1 wt. % propylene glycol, and more preferably 0 wt. % or no propylene glycol.

The compositions of the present invention can include one or more emollients. Emollients can, e.g., soften and/or soothe skin. Suitable emollients can include, for example, fatty alcohols such as, e.g., cetyl alcohol, stearyl alcohol, and combinations thereof. Fatty alcohols can sometimes be used to thicken compositions as well as provide emollient properties. Applicants have surprisingly found that compositions of the present invention, e.g., 5% fatty alcohol/1% Salcare® SC 96 and 1% fatty alcohol/5% Salcare® SC 96, exhibit roughly the same emulsion properties (e.g., viscosity, rheology, yield value, appearance, color). This was unexpected at cetyl alcohol concentrations down to only about 1 wt. %. Furthermore, it has been found that the emollient concentration can be reduced to about 1 wt. % or less in a composition with 2 wt. % PECs where the composition also has from about 3 wt. % to about 5 wt. % of a thickening agent. The wt. % range of fatty alcohols can be expanded when the concentration of PECs is lower. Even with a low concentration of emollient fatty alcohols, PEC compositions have been found to still provide an acceptable sensory experience. This is unexpected in that most hair care compositions, especially conditioners, have higher concentrations of emollient fatty alcohols to provide a sensory experience for the consumer. Also, coupled with the thickening agent, the PEC composition retains an acceptable viscosity. Additionally, the combination of a low concentration of emollient fatty alcohol and PECs with from about 3 wt. % to about 5 wt. % thickening agent provides for a rich, white opacity not observed with any of these ingredients individually.

[0046] The concentration of the emollient in the compositions of the present invention desirably can be tailored based on the concentration of thickening agent. With the thickening agent at 3-5 wt. %, for example, the emollient concentration can range, *e.g.*, from zero to less than about 1 wt. %, zero to about 0.9 wt. %, zero to about 0.8 wt. %, zero to about 0.7 wt. %, zero to about 0.6 wt. %, zero to about 0.5 wt. %, zero to about 0.4 wt. %, zero to about 0.3 wt. %, zero to about 0.2 wt. %, zero to about 0.1 wt. %, 0.1 wt. % to less than about 1 wt. %,

0.1 wt. % to about 0.9 wt. %, 0.1 wt. % to about 0.8 wt. %, 0.1 wt. % to about 0.7 wt. %, 0.1 wt. % to about 0.6 wt. %, 0.1 wt. % to about 0.5 wt. %, 0.1 wt. % to about 0.4 wt. %, 0.1 wt. % to about 0.3 wt. %, 0.1 wt. % to about 0.2 wt. %, 0.2 wt. % to less than about 1 wt. %, 0.2 wt. % to about 0.9 wt. %, 0.2 wt. % to about 0.8 wt. %, 0.2 wt. % to about 0.7 wt. %, 0.2 wt. % to about 0.6 wt. %, 0.2 wt. % to about 0.5 wt. %, 0.2 wt. % to about 0.4 wt. %, 0.2 wt. % to about 0.3 wt. %, 0.3 wt. % to less than about 1 wt. %, 0.3 wt. % to about 0.9 wt. %, 0.3 wt. % to about 0.8 wt. %, 0.3 wt. % to about 0.7 wt. %, 0.3 wt. % to about 0.6 wt. %, 0.3 wt. % to about 0.5 wt. %, 0.3 wt. % to about 0.4 wt. %, 0.4 wt. % to less than about 1 wt. %, 0.4 wt. % to about 0.9 wt. %, 0.4 wt. % to about 0.8 wt. %, 0.4 wt. % to about 0.7 wt. %, 0.4 wt. % to about 0.6 wt. %, 0.4 wt. % to about 0.5 wt. %, 0.5 wt. % to less than about 1 wt. %, 0.5 wt. % to about 0.9 wt. %, 0.5 wt. % to about 0.8 wt. %, 0.5 wt. % to about 0.7 wt. %, 0.5 wt. % to about 0.6 wt. %, 0.6 wt. % to less than about 1 wt. %, 0.6 wt. % to about 0.9 wt. %, 0.6 wt. % to about 0.8 wt. %, 0.6 wt. % to about 0.7 wt. %, 0.7 wt. % to less than about 1 wt. %, 0.7 wt. % to about 0.9 wt. %, 0.7 wt. % to about 0.8 wt. %, or 0.8 wt. % to less than about 1 wt. %, 0.8 wt. % to about 0.9 wt. %. Thus, the concentration of emollient can have a range bounded by any two of the aforementioned endpoints. Alternatively, with the thickening agent at 1-5 wt. %, for example, the emollient concentration can be, e.g., from about 5 wt. % or greater, about 6 wt. % or greater, about 7 wt. % or greater, about 8 wt. % or greater, about 9 wt. % or greater, about 10 wt. % or greater, about 20 wt. % or greater, about 30 wt. % or greater, about 40 wt. % or greater, or about 50 wt. % or greater.

[0047] Some PECs are described in U.S. Patent Application Publication Nos. 2005/0089494 and 2006/0251603, both of which are incorporated herein by reference in their entireties. 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.

[0048] 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.

[0049] 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 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.

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/polyquat-28 PECs, a pH range of from about 6.5 to about 7.5 may be used for PEC formation. The suitable pH can range from about 6.5 to about 7.5, from about 6.6 to about 7.5, from about 6.7 to about 7.5, from about 7.6, from about 7.7, from about 7.8, from about 7.9, from about

7.4, from about 7.3 to about 7.4, from about 6.5 to about 7.3, from about 6.6 to about 7.3, from about 6.7 to about 7.3, from about 6.8 to about 7.3, from about 6.9 to about 7.3, from about 6.5 to about 7.3, from about 7.2 to about 7.3, from about 6.5 to about 7.2, from about 6.6 to about 7.2, from about 6.7 to about 7.2, from about 6.8 to about 7.2, from about 6.9 to about 7.2, from about 7.1, from about 7.1, from about 6.5 to about 7.1, from about 6.6 to about 7.1, from about 6.7 to about 7.1, from about 6.8 to about 7.1, from about 6.9 to about 7.1, from about 7.0 to about 7.0, from about 6.5 to about 7.0, from about 6.7 to about 7.0, from about 6.8 to about 7.0, from about 6.7 to about 7.0, from about 6.8 to about 7.0, from about 6.9 to about 7.0, from about 6.9, from about 6.5 to about 6.9, from about 6.5 to about 6.9, from about 6.5 to about 6.7, from about 6.8 to about 6.7, or from about 6.8 to about 6.8, from about 6.5 to about 6.5 to about 6.7, or from about 6.5 to about 6.6. Thus, the pH can have a range bounded by any two of the aforementioned endpoints.

[0051] 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. %, from about 1 wt. % to about 30 wt. %, from about 2 wt. % to about 30 wt. %, from about 3 wt. % to about 30 wt. %, from about 4 wt. % to about 30 wt. %, from about 5 wt. % to about 30 wt. %, from about 10 wt. % to about 30 wt. %, from about 20 wt. % to about 30 wt. %, 0.1 wt. % to about 20 wt. %, from about 1 wt. % to about 20 wt. %, from about 2 wt. % to about 20 wt. %, from about 3 wt. % to about 20 wt. %, from about 4 wt. % to about 20 wt. %, from about 5 wt. % to about 20 wt. %, from about 10 wt. % to about 20 wt. %, 0.1 wt. % to about 10 wt. %, from about 1 wt. % to about 10 wt. %, from about 2 wt. % to about 10 wt. %, from about 3 wt. % to about 10 wt. %, from about 4 wt. % to about 10 wt. %, from about 5 wt. % to about 10 wt. %, 0.1 wt. % to about 5 wt. %, from about 1 wt. % to about 5 wt. %, from about 2 wt. % to about 5 wt. %, from about 3 wt. % to about 5 wt. %, from about 4 wt. % to about 30 wt. %, 0.1 wt. % to about 4 wt. %, from about 1 wt. % to about 4 wt. %, from about 2 wt. % to about 4 wt. %, from about 3 wt. % to about 4 wt. %, 0.1 wt. % to about 3 wt. %, from about 1 wt. % to about 3 wt. %, from about 2 wt. % to about 3 wt. %, 0.1 wt. % to about 2 wt. %, from about 1 wt. % to about 2 wt. %, or 0.1 wt. % to about 1 wt. %. Thus, the concentration of neutralizer can have a range bounded by any two of the aforementioned

endpoints. 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.

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 film-formation 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 film-formation assists in adhering the ends together.

[0053] 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, from about 0.5 micrometer to about 30 micrometers, from about 0.5 micrometer to about 20 micrometers, from about 0.5 micrometer to about 10 micrometers, from about 0.5 micrometer to about 5 micrometers, from about 0.5 micrometer to about 3 micrometers, from about 0.5 micrometer to about 2 micrometers, from about 0.5 micrometer to about 1 micrometers, from about 1 micrometer to about 40 micrometers, from about 1 micrometer to about 30 micrometers, from about 1 micrometer to about 20 micrometers, from about 1 micrometer to about 10 micrometers, from about 1 micrometer to about 5 micrometers, from about 1 micrometer to about 3 micrometers, from about 1 micrometer to about 2 micrometers, from about 2 micrometers to about 40 micrometers, from about 2 micrometers to about 30 micrometers, from about 2 micrometers to about 20 micrometers, from about 2 micrometers to about 10 micrometers, from about 2 micrometers to about 5 micrometers, from about 2 micrometers to about 3 micrometers, from about 3 micrometers to about 40 micrometers, from about 3 micrometers to about 30 micrometers, from about 3 micrometers to about 20 micrometers, from about 3 micrometers

to about 10 micrometers, from about 3 micrometers to about 5 micrometers, from about 5 micrometers to about 40 micrometers, from about 5 micrometers to about 30 micrometers, from about 5 micrometers to about 10 micrometers to about 10 micrometers, from about 10 micrometers, from about 10 micrometers to about 30 micrometers, from about 20 micrometers, from about 20 micrometers, from about 20 micrometers to about 30 micrometers, from about 20 micrometers to about 30 micrometers, or from about 30 micrometers to about 40 micrometers. Thus, the average PEC particle size distribution can have a range bounded by any two of the aforementioned endpoints. In some embodiments, the compositions of the present invention have a PEC particle distribution with an average PEC particle size of about  $5 \pm 3$  micrometers. The compositions of the present invention also can include PEC particles that exist within an interlocking microgel structure.

The compositions of the present invention can incorporate PECs at higher [0054] 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 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 1% to about 8% based on the total weight of the composition. The compositions of the present invention may contain the polyelectrolyte complex is in a concentration of from about 2% to about 4% based on the total weight of the composition. The concentration of PECs may be from about 0.1% to about 8%, from about 0.1% to about 7%, from about 0.1% to about 6%, from about 0.1% to about 5%, from about 0.1% to about 4%, from about 0.1% to about 3%, from about 0.1% to about 2%, from about 0.1% to about 1%, from about 0.1% to about 0.5%, from about 0.5% to about 8%, from about 0.5% to about 7%, from about 0.5% to about 6%, from about 0.5% to about 5%, from about 0.5% to about 4%, from about 0.5% to about 3%, from about 0.5% to about 2%, from about 0.5% to about 1%, 1% to about 8%, from about 1% to about 7%, from about 1% to about 6%, from about 1% to about 5%, from about 1% to about 4%, from about 1% to about 3%, from about 1% to about 2%, 2% to about 8%, from about 2% to about 7%, from about 2% to about 6%, from about 2% to about 5%, from about 2% to about 4%, from about 2% to about 3%, 3% to about 8%, from about 3% to about 7%, from about 3% to about 6%, from about 3% to about 5%, from about 3% to about 4%, 4% to about 8%, from about 4% to about 7%, from about 4% to about 6%, from about 4% to about 5%, 5% to about 8%, from about 5% to about

7%, from about 5% to about 6%, 6% to about 8%, from about 6% to about 7%, or 7% to about 8%. Thus, the concentration of PECs can have a range bounded by any two of the aforementioned endpoints.

[0055] PEC formation may be confirmed using any suitable method. An example of a suitable method includes microscopy. Under a light microscope, the structure of a polymer-polymer system can be investigated, and the sizes of any particles present can be determined using sizing software. Such a method can yield the particle size distribution of PECs, provided the presence of other ingredients does not obscure the PEC structure. Another method includes determining the particle size distribution using a Malvern particle size analyzer. Also, the physical properties of a composition of only PECs in a carrier solvent can indicate the presence of the PECs. For example, as the presence of PECs increases, such a composition increases in haziness and opacity. Also, the viscosity of such a composition decreases to a minimum that corresponds to the presence of the greatest amount of PECs for a given polymer-polymer system, where the PECs are the most compact.

[0056] The compositions of the present invention may be in the form of, *e.g.*, shampoos, conditioners, gels, rinses, emulsions (oil-in-water, water-in-oil or multiphase), lotions, creams, crèmes, ointments, pomades, sprays (pressurized or non-pressurized), spritzes, mousses, foams, shampoos, solutions, and solids (*e.g.*, as sticks, semisolids and the like). Thus, the hair care compositions of the present invention can include other components that may be suitable for use in these types of compositions. Preferably such components are compatible with PECs, in that the components do not disrupt PEC structure.

[0057] 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. One of ordinary skill in the art would recognize that the ingredients identified herein may have multiple properties and thus may fit within multiple ingredient categories, especially if it is a commercially available product with multiple components.

[0058] The aqueous carrier can include any suitable quantity of water, e.g., from about 25 wt% to about 97 wt% water (e.g., from about 30% to about 95% water). The composition of the present invention can include from about 30 wt. % to about 97 wt. % water, from about 50 wt. % to about 80 wt. % water, or from about 60 wt. % to about 70 wt. % water. The

water used in the composition of the present invention can be deionized water.

[0059] Suitable emulsifiers can include, for example, PPG-3 benzyl ether myristate (*e.g.*, Crodamol STS; Croda, Inc., Edison, NJ, USA), arachidyl alcohol and behenyl alcohol and arachidyl glucoside (*e.g.*, Montanov<sup>®</sup> 202; SEPPIC, Paris, France), polyacrylamide and C13-14 isoparaffin and laureth-7 (*e.g.*, Sepigel<sup>TM</sup> 305; SEPPIC), glyceryl stearate, isoceteth-20, oleth-2, methoxy PEG/PPG-7/3 aminopropyl dimethicone, PEG/PPG-18/18 Dimethicone (*e.g.*, DC-190; Dow Corning), PEG-12 dimethicone, PEG-40 hydrogenated castor oil, PEG-6 caprylic/capric triglyceride, aminomethyl propanol (AMP-95), and polyglyceryl-3 distearate.

**[0060]** Suitable surfactants, *e.g.*, detersive and/or cleansing surfactants, may be used, including, for example, disodium laureth sulfosuccinate, sodium laureth sulfate, polysorbate 20, polysorbate 60, cocamidopropyl betaine, and stearalkonium chloride. Such surfactants may be present in a shampoo composition. A conditioner preferably is substantially free of detersive surfactants, unless the conditioner is a two-in-one combination of a shampoo and conditioner. As used herein, "substantially free of detersive surfactants" means that the composition of the present invention contains less than about 2 wt. % detersive surfactants and more preferably less than about 1 wt. %, *e.g.*, 0 wt. % or no detersive surfactants.

[0061] Suitable conditioning agents can include, for example, tertiary or quaternary amines such as stearamidopropyl dimethylamine, polyquaternium-47 (*e.g.*, Merquat<sup>®</sup> 2001; Nalco, Naperville, IL, USA), silicone quaternium-22 (*e.g.*, Abil<sup>®</sup> T Quat 60; Evonik, Essen, Germany), cocamidopropyl betaine, polyquaternium-11, polyquaternium-39, polyquaternium-4 (Celquat<sup>®</sup> H-100; Akzo Nobel, Amsterdam, Netherlands), polyquaternium-4 (Celquat<sup>®</sup> L-200; Akzo Nobel) to 0.5 wt. %, polyquaternium-7, polyquaternium 10, and quaternium-80. Other suitable conditioning agents include, *e.g.*, Aqu D 4272 N-Hance SP100 (Acrylamidopropoyltrimonium Chloride/Acrylamide Copolymer), Distearoylethyl Dimonium Chloride; Cetearyl Alcohol (Varisoft EQ 65), Rhodia Guar S, Behenyltrimethyl Ammonium Chloride, and Behentimonium Chloride/Cetearyl Alcohol.

[0062] Although not essential, an effective amount of at least one preservative may be incorporated in the composition. For example, the preservative can be selected to kill bacteria that might otherwise be sustained or multiply in the composition. Suitable preservatives can include, for example, DMDM hydantoin, disodium EDTA, Kathon<sup>TM</sup> CG (actives: 5-chloro-2-methyl-4-isothiazolin-3-one and 2-methyl-4-isothiazolin-3-one) and the like, and combinations thereof.

[0063] Suitable gelling agents can be used. Suitable gelling agents can include, for

example, acrylic acid/VP crosspolymer (e.g., Ultrathix<sup>TM</sup> P-100; ISP).

[0064] Suitable silicones can be added, *e.g.*, to improve conditioning of hair. Suitable silicones can include, for example, polysiloxanes, including dimethicone, dimethiconol, cyclopentasiloxane, and cyclohexasiloxane. These include, *e.g.*, Abil<sup>®</sup> OSW 5 (84% Cyclopentasiloxane, 15% Dimethiconol, 1% Dimethicone; Evonik, Essen, Germany), DC-200 (dimethicone; Dow Corning, Midland, MI, USA), Fluid Blend (80% cyclomethicone, 20% dimethicone), and DC-1501 (D5/Dimethiconol; Dow Corning). Other suitable silicones include, for example, DC-949 (60% amodimethicone, 5% trideceth-12, 5% cetrimonium chloride, 30% water; Dow Corning), DC-8500 (82% Bis(C13-15 Alkoxy) PG-Amodimethicone, 18% C14-15 Alcohols; Dow Corning), aminopropyl dimethicone, DC 5-7113 Silicone Quat Microemulsion (66% Water, 25% Silicone Quaternium-16, 7.4% Undeceth-11, 4.5% Butyloctanol, 3.5% Undeceth-5; Dow Corning), DC-556 (phenyl trimethicone; Dow Corning), DC 2-8566 (amodimethicone; Dow Corning), and amodimethicone (ADM 1100; Wacker).

**[0065]** Suitable hair fixative may be used. Suitable examples include, for example VP/methacrylamide/vinyl imidazole Copolymer (*e.g.*, LUVISET<sup>®</sup> Clear; BASF), acrylic acid/vinylpyrrolidone crosspolymer (*e.g.*, Ultrathix<sup>TM</sup> P-100; ISP), vinyl caprolactum/VP/dimethylaminoethyl methacrylate copolymer (Advantage<sup>®</sup> S; ISP), and VP/VA Copolymer.

[0066] Additional ingredients may be added, including, for example, Cocos nucifera (coconut) oil, mineral oil (paraffinum liquidum), sodium chloride, hydrolyzed silk, keratin amino acids, ascorbic acid, panthenol, tocopherol acetate, biotin, niacinamide, triethanolamine, triticum vulgare (wheat) flour lipids, ceramide 3, persea gratissima (avocado) oil, tetrahexyldecyl ascorbate, glycine soja (soybean) sterols, alpha-glucan oligosaccharide, ethylhexyl methoxycinnamate, butyl methoxydibenzoylmethane, and tocopheryl acetate. A propellant may be used, including isobutane and propane and 152A (78.5% A46; 28.5% HFC 152A). Fatty acids can be used, such as myristic acid, palmitic acid, and stearic acid.

**[0067]** Additional components and/or ingredients can be provided to impart beneficial properties to the hair, *e.g.*, components and/or ingredients for imparting softness to the hair after drying, providing moisturization to the hair, providing pleasing consistency/viscosity of the composition, and providing volume/fullness to the hair.

[0068] 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. Such a complex can be manufactured in a side tank and added to the main batch tank after an emulsion phase is prepared and when the emulsion phase has cooled to 45-65 °C. A tertiary or quaternary amine at approximately 1-2 wt. % can be included in the emulsion phase and neutralized to a pH of about 4.0-6.0. Fatty alcohol (e.g., cetyl and/or stearyl) can be included at approximately 3-7 wt. % in conjunction with a low-HLB co-emulsifier such as glyceryl monostearate, distearate, or the like, in a 5:1 ratio and can be included in the emulsion phase. Silicones at, e.g., 0.1-10 wt. %, or a blend of dimethicones, cylcomethicones, phenyl and/or amino functional silicones also can be included. An emollient ester, such as PPG-3 benzyl ether myristate (e.g., Crodamol STS), at, e.g., 1-4 wt. % can be added to enhance product spreadability in addition to enhancing shine and hair feel. Propylene glycol at 10% can be included to improve freeze/thaw stability. Salcare® SC-96 can also be included, which can be after the emulsion phase and when the batch has cooled to 45-65 °C, at, e.g., 0.1-5 wt. % to increase viscosity, improve conditioned feel in hair, and improve long term stability and viscosity control. Another embodiment of a composition of the present invention can include, for example, the same composition except with approximately 1% fatty alcohol, no emollient ester, and Salcare® SC 96 at, e.g., 1-7 wt. %. An embodiment of a composition with approximately 3-7 wt. % fatty alcohol can include PEC complex that has been made first in a main tank, with the rest of the composition then made in the same tank, where Salcare<sup>®</sup> SC-96 has been increased to, e.g., 1-7 wt. %. An embodiment of a composition with approximately 1 wt. % fatty alcohol can include PEC complex that has been made first in a main tank, with the rest of the composition then made in the same tank, where Salcare® SC-96 has been increased to, e.g., 3-10 wt. %.

[0069] The present invention also provides embodiments of hair care formulations and their method of use that do not: increase the force required to comb hair, as compared to untreated hair; increase the force required to separate the adhesion of two hair fibers, as compared to untreated hair; increase the "three point bending" force, as compared to untreated hair; increase Young's Modulus, as compared to untreated hair; reduce the luster/shine of hair, as compared to untreated hair; or impart other quantifiable "negative" attributes.

[0070] 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.

[0071] The freeze-thaw stability of a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer may be improved by adding to the hair care composition from about 1% to about 5% of a thickening agent, wherein the percentage is based on the total weight of the composition. The thickening agent can include a quaternary ammonium nitrogen compound or polymer, *e.g.*, polyquaternium-37, or a mixture of polyquaternium-37 with propylene glycol dicaprylate/dicaprate and polypropylene glycol-1 trideceth-6. Alternatively, the freeze-thaw stability of a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer may be improved by adding to the hair care composition from about 10% or greater of propylene glycol, wherein the percentage is based on the total weight of the composition.

[0072] The methods of the present invention allow for the production of PECs in one container. Accordingly, one aspect of the present invention provides a method of producing a polyelectrolyte complex, the method comprising hydrating a methylvinylether/maleic acid copolymer having repeating units of the formula (Formula I)

in a container; reacting the methylvinylether/maleic acid copolymer with a base such as, *e.g.*, sodium hydroxide, in the container in an amount sufficient to at least partially neutralize the acidic groups thereof, to form an intermediate mixture; heating the intermediate mixture in the container; adding to the intermediate mixture in the container a cationic copolymer of a vinylpyrrolidone of the formula (Formula II)

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and a methacrylamidopropyl trimethylammonium chloride of the formula (Formula III)

mixing the polymers in the container at high shear force and at a temperature of at least about 50-60 °C to produce the polyelectrolyte complex.

Additionally, another aspect of the present invention allows for production of a [0073] hair care composition by combining the PECs produced in accordance with the present invention with other hair benefit agents. In accordance with the process of the present invention, such hair care compositions can be prepared by combining the PECs produced in accordance with the present invention with other hair benefit agents in a single container. Accordingly, the present invention also provides a method of producing a hair care composition comprising producing a polyelectrolyte complex, comprising the steps of hydrating a methylvinylether/maleic acid copolymer having repeating units of the formula I in a container; reacting the methylvinylether/maleic acid copolymer with a base such as, e.g., sodium hydroxide (e.g., a 20% sodium hydroxide solution, e.g., in water), in the container in an amount sufficient to at least partially neutralize the acidic groups thereof (e.g., by adding sufficient base to achieve a pH of from about 6.9 to about 7.0), to form an intermediate mixture; heating the intermediate mixture in the container, e.g., to a temperature of from about 50 °C to about 60 °C; adding to the intermediate mixture in the container a cationic copolymer of a vinylpyrrolidone of the formula II and a methacrylamidopropyl trimethylammonium chloride of the formula III; mixing the polymers in the container at high shear force, e.g., at a temperature of about 50-60 °C to produce the polyelectrolyte complex. The process of the present invention optionally includes further adding to the polyelectrolyte

complex in the container one or more additional components selected from aqueous carriers, surfactants, fragrances, conditioning agents, emollients, emulsifiers, thickening agents, preservatives, gelling agents, silicones, hair fixatives, humectants and moisturizers; and mixing the resulting composition in the container.

The present invention also allows for producing a hair care composition in two containers, the PECs being produced in one container and the rest of the composition being produced in a second container, where the PECs and other ingredients are then combined. In this aspect, the present invention also provides a method of producing a hair care composition comprising producing a polyelectrolyte complex between a methylvinylether/maleic acid copolymer and a cationic copolymer of a vinylpyrrolidone and a methacrylamidopropyl trimethylammonium chloride, comprising the steps of hydrating a methylvinylether/maleic acid copolymer having repeating units of the formula I in a first container; reacting the methylvinylether/maleic acid copolymer with a base such as, e.g., sodium hydroxide, in the first container in an amount sufficient to at least partially neutralize the acidic groups thereof, to form an intermediate mixture; heating the intermediate mixture in the first container; adding to the intermediate mixture in the first container a cationic copolymer of a vinylpyrrolidone of the formula II and a methacrylamidopropyl trimethylammonium chloride of the formula III; mixing the polymers in the first container at high shear force and at a temperature of at least about 50-60 °C to produce the polyelectrolyte complex; mixing in a second container one or more components selected from aqueous carriers, surfactants, fragrances, conditioning agents, emollients, emulsifiers, thickening agents, preservatives, gelling agents, silicones, hair fixatives, humectants and moisturizers; and mixing together in the first or second container the polyelectrolyte complex of the first container and the mixed components of the second container.

[0075] Phase diagrams, as indicated above, can be produced to determine the conditions under which PECs form, including within which polymer:polymer weight ratios and charge-charge ratios. Additional conditions may be varied as well, including temperature, solvent system, etc. Each of these conditions may be varied to determine their effects on PEC formation. Thus, due to the various conditions required to produce PECs and the vast number of variables involved, the production of PECs can be complicated and difficult. This is especially the case when a particular particle size is desired, as in producing PVMMA/polyquat-28 PECs that can mend split-end damaged hair. In addition to the polymer:polymer weight ratios and/or charge-charge ratios, etc., the particular production

method used also may impact the average particle size distribution of PECs. For example, the mixing speed and/or mixing time during the addition of one polymer to another, and the mixing speed and/or mixing time after this addition, can affect the PEC particle size distribution. Other process variables such as temperature, pH, and the presence of other charged compounds during the formation of PECs can also have an affect. The compositions of the present invention can be produced by methods that allow for production of PECs on a large scale where the proper particle size distribution for mending split-ends is achieved. The formation of PECs may be facilitated by application of heat at a certain stage, e.g., when the anionic polymer is neutralized with a base and/or when the cationic polymer is added to the anionic polymer to produce the complex. A composition may be preheated prior to combining polymers, may be heated with one or a subset of polymers, or may be heated during or after combining all polymers for PEC formation. For PVMMA/polyquat-28 PECs, it has been found that heating neutralized PVMMA prior to addition of the polyquat-28 is advantageous, e.g., with the temperature ranging from at least 50 °C to 60 °C or greater. The temperature may range from about 50 °C to about 60 °C, from about 51 °C to about 60 °C, from about 52 °C to about 60 °C, from about 53 °C to about 60 °C, from about 54 °C to about 60 °C, from about 55 °C to about 60 °C, from about 56 °C to about 60 °C, from about 57 °C to about 60 °C, from about 58 °C to about 60 °C, from about 59 °C to about 60 °C, from about 50 °C to about 59 °C, from about 51 °C to about 59 °C, from about 52 °C to about 59 °C, from about 53 °C to about 59 °C, from about 54 °C to about 59 °C, from about 55 °C to about 59 °C, from about 56 °C to about 59 °C, from about 57 °C to about 59 °C, from about 58 °C to about 59 °C, from about 50 °C to about 58 °C, from about 51 °C to about 58 °C, from about 52 °C to about 58 °C, from about 53 °C to about 58 °C, from about 54 °C to about 58 °C, from about 55 °C to about 58 °C, from about 56 °C to about 58 °C, from about 57 °C to about 58 °C, from about 50 °C to about 57 °C, from about 51 °C to about 57 °C, from about 52 °C to about 57 °C, from about 53 °C to about 57 °C, from about 54 °C to about 57 °C, from about 55 °C to about 57 °C, from about 56 °C to about 57 °C, from about 50 °C to about 56 °C, from about 51 °C to about 56 °C, from about 52 °C to about 56 °C, from about 53 °C to about 56 °C, from about 54 °C to about 56 °C, from about 55 °C to about 56 °C, from about 50 °C to about 55 °C, from about 51 °C to about 55 °C, from about 52 °C to about 55 °C, from about 53 °C to about 55 °C, from about 54 °C to about 55 °C, from about 50 °C to about 54 °C, from about 51 °C to about 54 °C, from about 52 °C to about 54 °C, from about 53 °C to about 54 °C, from about 50 °C to about 53 °C, from about 51 °C to about 53

°C, from about 52 °C to about 53 °C, from about 50 °C to about 52 °C, from about 51 °C to about 52 °C, or from about 50 °C to about 51 °C. Thus, the temperature can have a range bounded by any two of the aforementioned endpoints.

[0077] The compositions of the present invention may be prepared in a single container or may be prepared in separate containers, *e.g.*, by preparing the PECs separately from other components prior to combining the PECs with the other components. The methods described herein produce PECs of the same size, morphology, and stability as conventional methods, yet does so with reduced numbers of containers.

The step of adding a cationic polymer to the intermediate mixture can include [0078] adding a solution, e.g., an aqueous solution, of the cationic polymer to the intermediate mixture. The cationic polymer can be added to the intermediate mixture at any suitable rate, which can include, e.g., from about 0.3 kg/min per minute to about 1.0 kg/min per minute (e.g., if the period of addition is 10 minutes, the rates would be from about 3 kg/min to about 10 kg/min), from about 0.4 kg/min per minute to about 1.0 kg/min per minute, from about 0.5 kg/min per minute to about 1.0 kg/min per minute, from about 0.6 kg/min per minute to about 1.0 kg/min per minute, from about 0.7 kg/min per minute to about 1.0 kg/min per minute, from about 0.8 kg/min per minute to about 1.0 kg/min per minute, from about 0.9 kg/min per minute to about 1.0 kg/min per minute, from about 0.3 kg/min per minute to about 0.9 kg/min per minute, from about 0.4 kg/min per minute to about 0.9 kg/min per minute, from about 0.5 kg/min per minute to about 0.9 kg/min per minute, from about 0.6 kg/min per minute to about 0.9 kg/min per minute, from about 0.7 kg/min per minute to about 0.9 kg/min per minute, from about 0.8 kg/min per minute to about 0.9 kg/min per minute, from about 0.3 kg/min per minute to about 0.8 kg/min per minute, from about 0.4 kg/min per minute to about 0.8 kg/min per minute, from about 0.5 kg/min per minute to about 0.8 kg/min per minute, from about 0.6 kg/min per minute to about 0.8 kg/min per minute, from about 0.7 kg/min per minute to about 0.8 kg/min per minute, from about 0.3 kg/min per minute to about 0.7 kg/min per minute, from about 0.4 kg/min per minute to about 0.7 kg/min per minute, from about 0.5 kg/min per minute to about 0.7 kg/min per minute, from about 0.6 kg/min per minute to about 0.7 kg/min per minute, from about 0.3 kg/min per minute to about 0.6 kg/min per minute, from about 0.4 kg/min per minute to about 0.6 kg/min per minute, from about 0.5 kg/min per minute to about 0.6 kg/min per minute, from about 0.3 kg/min per minute to about 0.5 kg/min per minute, from about 0.4 kg/min per minute to about 0.5 kg/min per minute, or from about 0.3 kg/min per minute to about 0.4 kg/min per minute. Thus, the addition can have a range bounded by

any two of the aforementioned endpoints.

[0079] The formation of PECs depends on the mixing speed and mixing duration when one polymer is added to another. A high shear force can be used to ensure complete mixing. However, depending on the polymer-polymer systems, the mixing speed may have to be adjusted for proper PEC formation. For PVMMA/polyquat-28 PECs, the polyquat-28 can be added to the PVMMA over a period of 5 minutes, 10 minutes, 20 minutes, 25 minutes, 30 minutes, 40 minutes, 50 minutes, or 60 minutes. The polyquat-28 can be added to the PVMMA over a period of 30 minutes. The rate of addition can be any suitable rate in which PECs are formed. The rate can be determined based on the time needed for the period of addition. For example, the rate can be based on the amount added per minute per minute of the period of addition. For PVMMA/polyquat-28 PECs, this can be from about 0.4 kg/min per minute to 1.0 kg/min per minute. Thus, if the period of addition is 10 minutes, the rates would be from about 4 kg/min to about 10 kg/min.

[0080] Some embodiments of the compositions of the present invention include compositions with the formulations of PVMMA and polyquat-28 as in Table 1.

[0081] 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

[0082] The present invention provides for mixing in additional ingredients after PEC formation, wherein the mixing is sufficient to allow adequate incorporation of the one or more components.

[0083] The present invention provides a one-container process for producing a PEC, which is highly efficient, cost effective, and eliminates the additional time and labor required to transfer materials from one tank to another as is done in conventional PEC production processes. Accordingly the process of the present invention is particularly suitable for manufacturing PECs and hair care compositions containing PECs on a commercial scale, *e.g.*, about 500 to about 10,000 gallons.

[0084] The process of the present invention further provides for the production of PEC

compositions (e.g., at 8 wt. %) at concentrations that substantially exceed the concentration of PEC compositions which have been produced conventionally. Increasing the concentration at which PECs are produced has several advantages. The higher concentration allows for a smaller pre-mix tank to be used in connection with combining the PECs with other ingredients to produce hair care products, e.g., conditioners. Also, PECs can be prepared and stored for future use, and less storage space is needed for a more concentrated stock. Furthermore, fewer quality control steps would be required in that one batch of PECs could be prepared and tested at a higher concentration instead of preparing multiple batches at lower concentrations, requiring additional testing. Additionally, if a PEC stock is used for preparing a hair care composition, the PEC stock will not require as much volume in a processing container, allowing for the addition of a greater volume of other ingredients and allowing for higher concentrations to be achieved for additional ingredients. For example, the Applicants have found for preparing PVMMA/polyquat-28 PECs, it is not necessary to dilute the polyquat-28 (e.g., Conditioneze NT-20 which is commercially available at a concentration of 20% actives). Rather, in accordance with the present invention, PVMMA/polyquat-28 PECs can be prepared in a single container by directly adding Conditioneze NT-20 at full strength to the PVMMA copolymer, which surprisingly produces a stable PEC at high concentration. Further, Applicants have found that the higher PEC concentration in the compositions produced in accordance with the present invention are substantially more likely than conventional PEC compositions of lower concentration to form stable emulsions with other ingredients used to produce hair care products such as, e.g., conditioners.

[0085] The present invention provides a method of graphically reconstructing the repair process in the mending of a split-end of a hair fiber. In one embodiment the method includes securing a hair fiber to a surface, wherein the hair fiber secured to the surface has a split-end or is damaged while on the surface to produce a split-end; associating the surface with a magnifying device, wherein the split-end is positioned within the field of view of the magnifying device; associating an image capture device with the magnifying device so as to capture an enlarged image of the field of view of the magnifying device; treating the split-end with a composition, wherein the composition repairs the split-end and wherein the split-end fuses together; and via the image capture device associated with the magnifying device, capturing an image of the repair of the split-end during treatment with the composition, and storing a representation of the captured image of the repair for subsequent retrieval. The

method of graphically reconstructing hair mending in accordance with the present invention includes using video to record the hair mending process in real-time. Desirably the hair mending process is recorded using a video device adapted to record the reparation process, in real-time, at a magnification sufficient to visualize the reparation process with the naked eye, *e.g.*, at 20-50X magnification. The video can be stored on a recording media, using methods that are well known to those of ordinary skill in the art, for subsequent retrieval.

[0086] Additionally, the methods of the present invention may include securing a hair fiber to a surface, wherein the hair fiber secured to the surface has a split-end or is damaged while on the surface to produce a split-end. The hair fiber, thus, may have a split-end prior to securing it to the surface, or a split-end can be induced (for example, by mechanical means) after the hair fiber is secured to the surface. The surface may be any surface that can be associated with a magnifying device. Such a surface can include, for example, a table top or a microscope slide. The properties of the surface can be determined by the type of magnifying device used. For example, if a light microscope is used wherein light is transmitted through a sample, a transparent surface, *e.g.*, a microscope slide, may be used. As another example, the surface may be a stage on which the hair fiber rests. As yet another example, if the magnifying device is a simple lens, such as a magnifying glass, the surface can be a table top. Nothing should interfere with the mending of the split-end. For example, cover slips for microscope slides should not be used if the slip would prevent the ends of the split-end from coming together.

[0087] The hair fiber can be secured by any suitable means, including, for example, using one or more clamps. The hair fiber can also be secured using tape, for example, when using a table top or a microscope slide. As long as the split of the hair fiber can be observed with the magnifying device, any suitable means may be used for securing (e.g., fastening) the hair fiber to the surface.

[0088] The methods of the present invention can include associating the surface with a magnifying device, wherein the split-end is positioned within the field of view of the magnifying device. The magnifying device may be, for example, a magnifying glass, a microscope (e.g., a light microscope). Each magnifying device has a field of view. "Field of view" means the image area that the magnifying devices magnifies for observation. The split-end is positioned within the field of view such that the split-end is magnified for observation.

[0089] The methods of the present invention can include associating an image capture

device with the magnifying device so as to capture an enlarged image of the field of view of the magnifying device. The image capture device can be, for example, a digital recorder. The image capture device also can be analog. Non-limiting examples of image capture devices include still cameras, video cameras, charge-coupled device (CCD) cameras, digital cameras and/or camcorders, or any other similar recording device.

[0090] The methods of the present invention can include treating the spilt-end with a composition, wherein the composition repairs the split-end and wherein the split-end fuses together. Treatment may include, for example, massaging the composition into the hair. Treatment also may include, for example, rinsing the hair to remove the composition, such as with a rinse-off composition. The composition may be allowed to remain on the hair, for example, when using a leave-in composition. The method of treatment may be any suitable method, including methods described herein. The composition may be any suitable compositions, including compositions described herein.

[0091] The methods of the present invention can include a step where, via the image capture device associated with a magnifying device, capturing an image of the repair of the split-end during treatment with the composition, and storing a representation of the captured image of the repair for subsequent retrieval. The image capture device can capture the repair of the split-end using any suitable means. For example, if the image capture device is a digital recorder, the digital recorder may generate an electronic file. As another example, if the image capture device is a camera, the camera may capture the repair using photographic film.

[0092] Storage of a representation of a captured image of repair for subsequent retrieval may be through any suitable means. For example, this may include storing an electronic representation of the image, for example, using an electronic file accessible on a digital camera and/or computer. This may also be through the use of photographic film.

[0093] Furthermore, the present invention can be used for any method of communicating to consumers. These may include, for example, the use of still photographs (e.g., before and after photographs); diagrams, depictions, and/or drawings; computer animation; and/or in-vivo/in-vitro/in-situ video using actual human hair fiber or analogues (e.g., sheep wool). Additionally, the methods of communication can be in print, on television, on the Internet, instore, in-person (e.g., live demonstrations), and/or on social media. The methods of the present invention also can be used for the explanation of a binding mechanism (e.g., PECs shrinking to draw together two ends of a split-end).

The present invention also provides a method of quantitatively assessing the [0094] degree of split-end hair fiber repair, the method comprising obtaining a tress of hair, preferably twice-bleached hair; producing at least one spilt-end of a hair fiber in the tress (e.g., by combing and/or blow drying the tress sufficiently to produce split-ends); removing at least one hair fiber having a split-end from the tress; optionally marking the at least one removed hair fiber; capturing a first image of the split-end of the at least one removed hair fiber; associating the hair fiber with the tress from which the hair fiber was removed; treating the tress and the hair fiber with a composition; removing the hair fiber from the tress; capturing a second image of the end of the hair fiber, wherein the end is the same end captured for the first image of the split-end of the at least one removed hair fiber; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair; and summing the assigned numbers for all marked hair fibers and dividing the sum by the total number of marked hair fibers. Exemplary compositions of the present invention, when assessed according to this method can achieve an average value of, e.g., 0.85 or greater, e.g., 0.925, 0.95, and even 0.975 based on an assessment of 20 fibers.

[0095] The methods of the present invention can include producing split-ends. This may include combing and blow drying the tress of hair to produce at least one hair fiber of the tress having a spilt-end. Other methods include selecting split-ends naturally. For example, hair samples may be attained from live specimens wherein the hairs are already split. Due to variety, however, this method may cause data to have poor reproducibility.

[0096] The methods of the present invention can include marking at least one removed hair fiber of the tress of hair. The split-end can be marked right at the beginning of the split. If the mark is gone after treatment with the composition, this can indicate that the split-end broke off the hair fiber. When assigning a value to the fibers, then, this fiber can be excluded since the split-end neither remained as a split-end nor was repaired. Thus, *e.g.*, false positives can be avoided when assessing the mending ability of a composition.

[0097] The methods of the present invention can include the capturing an image of the split-end of at least one removed hair fiber. Examples of such capturing methods are described herein. This step may include storing an electronic representation of the image, for example, using an electronic file accessible on a digital camera and/or computer.

[0098] The methods of the present invention can include associating the at least one marked hair fiber with the tress from which the hair fiber was removed. Replacing the removed hair fiber back with the tress provides a more natural environment for testing the

mending ability of a composition. The hair fibers with split-ends remain with other hair fibers that may not have split-ends and remain within a larger grouping of hair fibers. This situation better simulates the environment of a normal lock of hair, *e.g.*, on a human head. Simply removing the hair fibers with split-ends and testing compositions on these removed fibers would only provide results based on an artificial environment. Thus, when using the method to test compositions for use by consumers, the present invention allows for reporting results that are meaningful to those consumers, *e.g.*, results that report on the mending of hair fibers that are in an environment important to the consumers.

[0099] The methods of the present invention can include treating the tress and the at least one marked hair fiber with a composition. Treatments may include, for example, massaging the composition into the hair. Treatment also may include, for example, rinsing the hair to remove the composition, such as with a rinse-off composition. The composition may be allowed to remain on the hair, for example, when using a leave-in composition. The method of treatment may be any suitable method, including methods described herein. The composition may be any suitable compositions, including compositions described herein.

[0100] The methods of the present invention can include determining parameters of hair mending. For example, the rate at which a split-end is mended can be determined under defined conditions, *e.g.*, those described herein. Compositions can then be compared to determine which provide for faster mending of split-ends. The split-end mending efficacy of the compositions used and/or tested also can be determined, *e.g.*, using the video methods described herein. Mending of a split-end is preferable within one hour, less than 50 minutes, less than 40 minutes, less than 30 minutes, less than 20 minutes, less than 10 minutes, less than 5 minutes, or less than 1 minute. The completion or amount of mending may be 10% or greater, 20% or greater, 30% or greater, 40% or greater, 50% or greater, 60% or greater, 70% or greater, 80% or greater, 90% or greater, or complete (*e.g.*, about100%). The mending can be measured against a negative control or no treatment, wherein the treated split-end can show a statistically significant improvement in split-end mending (*e.g.*, faster mending and/or greater amount or completion of mending).

[0101] The methods of the present invention can include assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair and summing the assigned numbers for all marked hair fibers and dividing the sum by the total number of marked hair fibers. Thus, e.g., when there is an accounting for all of the split-ends of a tress, the resulting value will report on the ability of the composition to mend split-ends. A composition with

poor mending ability will have a value that is low and/or approaches zero, whereas a composition with better mending ability will have a value that is higher and/or approaches one. Compositions thus may be compared using the method of the present invention where a composition that results in a higher value provides better mending than a composition that results in a lower value.

[0102] Any type of split-end can be mended by the methods of the present invention. One type of split-end includes, for example, the primary split-end. With this type of splitend, the hair fiber contains a single split along the longitudinal axis of the hair fiber, producing two ends of the hair fiber. Another example of a split-end includes the double Y split-end, which is created by an additional split along the longitudinal axis of an end of a primary split. The tree split-end is an example of a split-end where there are several double Y splits within one hair fiber. Another example of a split-end is where the end of a hair fiber has many short splits that feather out, often resulting in fiber shafts of varying thickness. Another example of a split-end is the long split-end where one side of a primary split end is significantly longer than the other (e.g., one side broke off). The taper split-end is another example of a split-end where only a portion of the hair fiber remains at the end. Split-ends also may, for example, be characterized by the number of ends, such as with the triple splitend with two splits of the hair fiber. A deep split-end is an example of a split that occurs at the mid-point of a hair shaft (not necessarily the end). A baby split-end is an example of a split-end that only has a small split in the hair fiber. The term "split-ends" as used herein also can include splits that do not extend to the end of the hair fiber. An example of such a split is an incomplete split. Figures 4A-4D show some types of split-ends.

[0103] The hair fibers used in the methods of the present invention may be of any mammalian hair. In one embodiment, the hair is human hair. Human hair of any nationality, region, or ethnicity may be used, e.g., European, Asian, African, or Caucasian. In an embodiment, the hair is European hair. The hair may have any type of damage due to, e.g., bleaching once, bleaching twice, bleaching more than twice, dyeing, or mechanical damage. The hair fibers may have no damage, for example, as with virgin hair. Additionally, the type of hair used in the methods of the present invention can be varied from one run of the method to another. This varying of the hair can correspond to the use and testing of various compositions, which can, e.g., depend on what type of results are desired to be communicated to consumers. Varying the hair type and/or composition will allow for the communication of results for any hair type and any hair care composition.

[0104] An aspect of the present invention includes selecting a hair fiber with a split end and cutting to, *e.g.*, about 1-1.5 inch in length (measuring from the split end side). A small piece of clear tape can be used to secure the fiber on, for example, a glass slide. The glass slide can be placed under a microscope with, *e.g.*, 50X magnification and that has an interface with a camera and computer. Lighting and focus can be adjusted accordingly. Depending on set-up, a recording can take place within the computer interface or a video camera can be set-up on, *e.g.*, a tripod pointing at the computer screen. With the camera recording, one drop of a composition, *e.g.*, containing 2% active complex can be applied over the split-end using a micropipette. Lighting and focus can be further adjusted accordingly. A blow drier can be turned on, *e.g.*, medium heat and aimed toward the split end. Recording can take place during and until the split end has fully or partially mended.

An aspect of the invention includes taking human hair, preferably damaged, most [0105] preferably twice-bleached European hair, and assembling into, e.g., wax bound strips that may be, for example, 1-1.5 inches wide. These tresses are then combed many thousands of times while a blow dryer is pointed perpendicularly to brushing to form an appropriate number of split- ends. A select number (for example, 10-20) of fibers with split-ends are selected and pulled out of the fiber assembly. Pictures of each split-end are taken with the use of a microscope at 20-50X magnification. Brass ferrule tabs are clamped on the end opposite of the split-end of each fiber. These brass tabs are then labeled and placed between two pieces of masking tape. The assembly is then inserted into the middle of the hair tress and a paper clamp is affixed to keep the assembly in place. The tress is then treated with a rinse-out split-end mending composition for a series of, e.g., three cycles. After the final cycle, the clamp is removed and new pictures of the split-ends are taken. By comparing the before and after pictures, an evaluator can give a value of "0" for no mending, "1/2" for partial mending, and "1" for complete mending. Summing these numbers and dividing by the total number of fibers allows for the expression of the degree of split-end repair.

[0106] One embodiment of the present invention provides a method of quantitatively assessing the degree of split-end hair fiber repair, the method comprising obtaining a tress of hair; producing at least one hair fiber of the tress to have a spilt-end; removing at least one hair fiber having a split-end from the tress; optionally marking the at least one hair fiber; capturing a first image of the split-end of the at least one hair fiber and storing a representation of the image; associating the hair fiber with the tress from which the hair fiber was removed; treating the tress and the hair fiber with a composition, wherein the

composition comprises a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, from about 1% to about 5% of a thickening agent, about 10% or greater of propylene glycol, and about 5% or greater of an emollient, wherein the percentages are based on the total weight of the composition; removing the hair fiber from the treated tress; capturing a second image of the end of the hair fiber removed from the treated tress; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair with respect to the hair fiber to assess the degree of split-end repair of the hair fiber; and if more than one hair fiber is assessed, optionally determining an average value with respect to the assessed hair fibers.

Another embodiment of the present invention provides a method of quantitatively [0107] assessing the degree of split-end hair fiber repair, the method comprising obtaining a tress of hair; producing at least one hair fiber of the tress to have a spilt-end; removing at least one hair fiber having a split-end from the tress; optionally marking the at least one hair fiber; capturing a first image of the split-end of the at least one hair fiber and storing a representation of the image; associating the hair fiber with the tress from which the hair fiber was removed; treating the tress and the hair fiber with a composition, wherein the composition comprises a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, from about 3% to about 5% of a thickening agent, and about 1% or less of an emollient, wherein the percentages are based on the total weight of the composition; removing the hair fiber from the treated tress; capturing a second image of the end of the hair fiber removed from the treated tress; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair with respect to the hair fiber to assess the degree of split-end repair of the hair fiber; and if more than one hair fiber is assessed, optionally determining an average value with respect to the assessed hair fibers.

[0108] Another embodiment of the present invention provides a method of quantitatively assessing the degree of split-end hair fiber repair, the method comprising obtaining a tress of hair; producing at least one hair fiber of the tress to have a spilt-end; removing at least one hair fiber having a split-end from the tress; optionally marking the at least one hair fiber; capturing a first image of the split-end of the at least one hair fiber and storing a representation of the image; associating the hair fiber with the tress from which the hair fiber was removed; treating the tress and the hair fiber with a composition, wherein the composition comprises a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, wherein the viscosity of the composition is greater

than 3000 cps; removing the hair fiber from the treated tress; capturing a second image of the end of the hair fiber removed from the treated tress; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair with respect to the hair fiber to assess the degree of split-end repair of the hair fiber; and if more than one hair fiber is assessed, optionally determining an average value with respect to the assessed hair fibers.

Another embodiment of the present invention provides a method of quantitatively assessing the degree of split-end hair fiber repair, the method comprising obtaining a tress of hair; producing at least one hair fiber of the tress to have a spilt-end; removing at least one hair fiber having a split-end from the tress; optionally marking the at least one hair fiber; capturing a first image of the split-end of the at least one hair fiber and storing a representation of the image; associating the hair fiber with the tress from which the hair fiber was removed; treating the tress and the hair fiber with a composition, wherein the composition comprises a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer in the form of an emulsion with improved freeze-thaw stability relative to comparative compositions containing such polyelectrolyte complexes; removing the hair fiber from the treated tress; capturing a second image of the end of the hair fiber removed from the treated tress; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair with respect to the hair fiber to assess the degree of split-end repair of the hair fiber; and if more than one hair fiber is assessed, optionally determining an average value with respect to the assessed hair fibers.

[0110] As noted above, an image capture system is used in an embodiment of the invention to produce a representation of the appearance of the split end both before and after repair or attempted repair. While various such image capture systems are possible within the scope of the teachings herein, an exemplary such system is shown in FIG. 6. In particular, FIG. 6 is a schematic diagram showing an image data capture system in which the described image capture and processing steps may be executed.

[0111] The illustrated system 600 comprises a number of interacting components, including a sample support device 601, such as a platform or surface suitable for supporting a sample 602. The sample support device 601 may be transparent to allow under-lighting, via a light source 603. The light source 603 may also be situated above the support device 601 to provide illumination via reflectance.

[0112] An electrical image capture device 604 is provided for capturing an enlarged image or video sequence of the sample 602 under treatment. In this regard, a magnification

device 605 is provided adjacent the sample support device 601, between the sample support device 601 and the image capture device 604. The image capture device 604 may be a still camera or video camera adapted to capture a sequence of images. The magnification device 605 may be an optical magnifying lens or lens arrangement such as an optical microscope, or may be an electronic magnification device such as an electron microscope or the like. In an embodiment of the invention, the image capture device 604 and/or the magnification device 605 may be scannable to capture images of different regions or segments of the sample 602.

- [0113] An auxiliary computing and data capture device 606 is provided from controlling and receiving data from the image capture device 604. The auxiliary computing and data capture device 606 is a com-putting a processing device able to receive electronic image data from the image capture device 604 and send control signals to the image capture device 604 to cause image capture. The auxiliary computing and data capture device 606 may also control the illumination device 603.
- **[0114]** In an embodiment of the invention, the auxiliary computing and data capture device 606 is communicably linked to an electronic database 607, either locally or remotely via a wired or wireless link. In this manner, the auxiliary computing and data capture device 606 is able to gather data from the image capture device 604 and to store the gathered data in the electronic database 607 for later analysis, manipulation, and/or retransmission, e.g., to a printing or publishing facility (not shown).
- [0115] The auxiliary computing and data capture device 606 may be a computer such as a person computer, laptop computer, workstation or the like, and operates by the computer-execution of computer readable instructions stored on a computer-readable medium. The computer-readable medium is a tangible medium such as a magnetic or optical disc system, flash drive, PRO, etc. Thus, the steps discussed herein that involve the capture, manipulation, and/or transmission of data are executed in this manner. A user interface 608 allows a user to receive information from the auxiliary computing and data capture device 606 in human understandable form and to provide input to the device in computer understandable form. In an embodiment of the invention, the user interface 608 includes a graphical user interface on a screen and a keyboard, keypad, stylus pad, mouse, and/or other user input mechanism.
- [0116] In one embodiment of the present invention, the image capture device is a video camera adapted to record reparation of one or more hair shafts, e.g., a hair shaft containing one or more split-ends, in real-time. Desirably, the video is recorded at magnification sufficient to visualize with the naked eye the hair reparation process in real-time. Suitable

magnification can include, e.g., 20-50X magnification.

[0117] The following examples further illustrate the invention but, of course, should not be construed as in any way limiting its scope.

#### EXAMPLE 1

[0118] This example demonstrates production of hair care compositions comprising a polyelectrolyte complex using a two-tank process.

[0119] Table 2 shows the ingredients.

[**0120**] Table 2

Ingredient	Composition 1A	Composition 1B	Composition 1C
Ingrodient	(Conditioner)	(Overnight	(Leave-in
	(wt. %)	Crème)	Crème)
	(₩6. 70)	(wt. %)	(wt. %)
Water	15.55	15.55	15.55
PVMMA Copolymer	0.2	0.2	0.2
Sodium Hydroxide (20%), Water	0.25	0.25	0.25
Polyquaternium-28 (20%), Water	9	9	9
Water	43.3997	48.7997	52.7997
Disodium EDTA (99.3%	0.05	0.05	0.05
EDTANa <sub>2</sub> *2H20 in water)			
Aspartic Acid	0.75	0.75	0.75
Stearamidopropyl Dimethylamine	2	2	2
Cetyl Alcohol	4	1	1
Stearyl Alcohol	1	0	0
Glyceryl Stearate	1	0.2	0.2
Aminopropyl Dimethicone, Water	0	0.5	0
PPG-3 Benzyl Ether Myristate	2	0	0
(Crodamol STS)			
Propylene Glycol	10	10	0.5
Water	4	4	8
Fluid Blend	5	0	0
Abil® OSW 5	0	2	0
Dimethicone	0	1	2
DC-8500	0	1	0
Parfum (Fragrance)	0.6	0.5	0.5
DMDM hydantoin, water	0.2	0.2	0.2
Cocos Nucifera (Coconut) Oil	0.0001	0.0001	0.0001
Keratin Amino Acids, Water	0.0001	0.0001	0.0001
Flower/Leaf extract	0.0001	0.0001	0.0001
Salcare® SC 96	1	3	3
Glycerin, USP VEG	0	0	1
Cyclopentasiloxane, Dimethiconol	0	0	1
Phenyl Trimethicone	0	0	2

- [0121] One of ordinary skill in the art would be able to determine the proper RPM settings for the mixers as listed in this Example based on the identity of the particular tanks to be used in following this protocol.
- [0122] In a suitable, stainless steel premix tank, charge water set at 113-131 °F (45-55 °C). Begin vigorous turbine mixing (turbine mixer at vigorous clockwise direction; side sweep mixer off). Once the batch temperature is 86 °F (30 °C) or above, and with vigorous mixing, slowly add PVMMA Copolymer (Gantrez® S-97 BF; ISP, Wayne, NJ, USA) to the premix tank. Adjust mixing accordingly in order to achieve vigorous agitation without creating excessive foam on the liquid surface. Once all of the powder has been added, use water set at 113-131 °F (45-55 °C) to rinse the mixing blades and side walls. Once the rinse is completed, turn on the side sweep agitator and begin heating to 122-131 °F (50-55 °C). (The powder lumps will disperse more quickly when the premix is 113-131 °F (45-55 °C)). Continue heating and maintain the temperature throughout the mix time. Set the mixer (turbine mixer at vigorous counterclockwise direction; side sweep mixer at moderate clockwise direction).
- [0123] Mix the batch for a minimum of 20 minutes or until no particles or lumps are present.
- [0124] Once the premix is uniform and free of lumps, reduce turbine mixing to moderate. Add the sodium hydroxide to the premix tank and mix for 10 minutes. The solution will have a low viscosity and will appear slightly hazy. (Mixer settings: turbine mixer at moderate counterclockwise direction; side sweep mixer at moderate clockwise direction.) Mix the batch for a minimum of 10 minutes or until completely uniform.
- [0125] Once the batch is uniform, submit a sample for an in-process pH test and approval to proceed. Continue to retest the sample until a stable pH reading of 6.9-7.0 is achieved. If pH is low, add additional sodium hydroxide 20% to meet the specification range. Remix and resample. If pH is high, add additional aspartic acid to meet specification range. Remix and resample.
- [0126] Once the Gantrez has been completely hydrated and the pH is within specification, ensure the premix temperature is 122-131 °F (50-55 °C). Heat if necessary. Once the premix is 122-131 °F (50-55 °C), turn off the side sweep agitator, reverse turbine direction and increase turbine mixing speed to create a vortex (turbine mixer at vigorous clockwise direction; side sweep mixer off).
- [0127] Slowly add the polyquaternium 28 (Conditioneze® NT-20 (20% active); ISP,

Wayne, NJ, USA) to the premix vessel. Add slowly to the premix vessel to ensure the proper polyelectrolyte complex particle size. Use a drum spout to regulate addition flow rate. Do not dump the entire open container into the premix vessel at once. During this material addition, the premix will become very viscous and begin to turn white. Once all of the material has been added, the solution will again thin out and appear milky in appearance.

- **[0128]** Reverse turbine direction and turn on side sweep agitator. Mix vigorously for 20 minutes or until the premix is completely uniform (turbine mixer at vigorous counterclockwise direction; side sweep mixer at moderate clockwise direction). Mix the batch for a minimum of 20 minutes or until no visible particles or lumps are present.
- [0129] Once the mix is complete, take a sample and visually inspect the premix. Ensure it is completely white and not hazy or gray. Also ensure the premix viscosity is thin and milk-like and not too viscous. If the premix is not thin and milky white, continue mixing vigorously for an additional 20 minutes and resample.
- [0130] Once the premix is completely uniform, reduce agitation and begin cooling to 86-113 °F (30-45 °C). Maintain mixing and temperature until the premix is ready to be added to the main compounding tank (turbine mixer at moderate counterclockwise direction; side sweep mixer at moderate clockwise direction).
- [0131] Into a main stainless steel compounding tank, charge water set at 180-185 °F (82-85 °C) with moderate mixing (turbine mixer at moderate counterclockwise direction; side sweep mixer at moderate clockwise direction). Slowly add the disodium EDTA and aspartic acid to the main compounding tank. After the items have been added, begin heating the batch to 180-185 °F (82-85 °C), as necessary (turbine mixer at moderate counterclockwise direction). Continue heating the batch to 180-185 °F (82-85 °C). Mix the batch for a minimum of 5 minutes or until the solids are completely dissolved.
- [0132] Once the batch temperature is 176 °F (80 °C) or above, add the stearamidopropyl dimethylamine to the main compounding tank and mix until it is completely melted (turbine mixer at moderate counterclockwise direction; side sweep mixer at moderate clockwise direction). Continue heating the batch to 180-185 °F (82-85 °C). Mix the batch for a minimum of 5 minutes or until the solids are completely melted.
- [0133] Once the batch temperature is 180-185 °F (82-85 °C), add the following items to the main compounding tank: cetyl alcohol, stearyl alcohol, glyceryl stearate, and PPG-3 benzyl ether myristate for Composition 1A (conditioner); cetyl alcohol, glyceryl stearate, and aminopropyl dimethicone for Composition 1B (overnight crème); cetyl alcohol and glyceryl

stearate for Composition 1C (leave-in crème). Once the batch temperature is again 180-185 °F (82-85 °C), maintain this temperature and mix for a minimum of 30 minutes (turbine mixer at moderate counterclockwise direction; side sweep mixer at moderate clockwise direction). Mix the batch while holding it between 180-185 °F (82-85 °C).

- [0134] Take a sample from the main compounding tank and visually verify that all of the solids have been melted and that the batch is smooth and uniform. If there remains any non-dissolved materials or particles, continue vigorous mixing and resample.
- [0135] Begin cooling the batch to 104-113 °F (40-45 °C) and reduce the turbine and side sweep agitator speeds to moderate/slow to help de-aerate the batch (turbine mixer at moderate counterclockwise direction; side sweep mixer at slow clockwise direction). While cooling and once the batch temperature is 167 °F (75 °C) or below, add the propylene glycol to the main compounding tank (turbine mixer at moderate counterclockwise direction; side sweep mixer at moderate clockwise direction). For Composition 1C (leave-in crème), also add glycerin at this mixing speed.
- [0136] Once the main batch temperature is 150 °F (65 °C) or below for Composition 1A (conditioner) or 131 °F (55 °C) or below for the crèmes turn off the side sweep agitator, reverse the direction of the turbine agitator, and increase mixing to create a slight vortex (turbine mixer at moderate clockwise direction; side sweep mixer off). Add the Salcare® SC-96 (65% polyquaternium-37, 25% propylene glycol dicaprylate/dicaprate, 10% PPG-1 trideceth-6; Ciba/BASF, Basel, Switzerland) to the main compounding tank and continue cooling to 104-113 °F (40-45 °C). (Mix Salcare® SC-96 well in its container prior to addition to the batch.)
- [0137] Once all of the material has been incorporated into the batch and is no longer sitting on the surface, reverse turbine agitator and turn on side sweep agitator (turbine mixer at moderate counterclockwise direction; side sweep mixer at moderate clockwise direction).
- [0138] Once the main batch temperature is 150 °F (65 °C) or below for Composition 1A (conditioner) or 131 °F (55 °C) or below for Composition 1B (overnight crème), and the premix is ready, add the premix to the main compounding tank. While the premix is being added, turn off the turbine mixer or reduce it to slow as the liquid level passes the top set of turbine blades to minimize aeration. Once the premix has been added, rinse the premix tank with water set at 77-95 °F (25-35 °C) and add it to the main compounding tank. The batch viscosity will decrease as the premix is mixed into the batch. Adjust mixer speeds accordingly to avoid splashing (turbine mixer at moderate counterclockwise direction; side

sweep mixer at moderate clockwise direction).

**[0139]** For Composition 1A (conditioner), mix the batch for a minimum of 15 minutes or until the batch is uniform. Once the main batch temperature is 122 °F (50 °C) or below, add the Fluid Blend directly to the main compounding tank. Mix well during addition. Increase mixing as necessary to mix in the silicone turbine mixer at vigorous counterclockwise direction; side sweep mixer at moderate clockwise direction).

[0140] For the crèmes, once all of the premix has been added, charge with water set at 77-95 °F (25-35 °C) below the surface of the batch, to minimize aeration, if possible. Mix the batch for a minimum of 15 minutes or until the batch is uniform. Continue cooling to 104-113 °F (40-45 °C). In a suitable stainless steel phase vessel or container, premix the following silicone items with vigorous agitation: Abil® OSW 5 (84% cyclopentasiloxane, 15% dimethiconol, 1% dimethicone), and dimethicone (60,000 cst) for Composition 1B (overnight crème); dimethicone (60,000 cst), phenyl trimethicone (DC-556; Dow Corning), and D5 dimethiconol (DC-1501) for Composition 1C (leave-in crème). Once the silicone premix is uniform, add it to the main compounding tank and allow the silicone premix container to drain out as much as possible into the main batch tank. Increase mixing as necessary to mix in the silicones (turbine mixer at vigorous counterclockwise direction; side sweep mixer at moderate clockwise direction).

- [0141] When the batch temperature is 113 °F (45 °C) or below, add the following ingredients to the main compounding tank: fragrance (parfum), DMDM hydantoin, cocos nucifera oil, keratin amino acids, flower/leaf extract. For the Composition 1B (overnight crème), also add DC-8500 (82% Bis(C13-15 alkoxy) PG-Amodimethicone, 18% C14-15 alcohols; Dow Corning, Midland, MI, USA). Continue mixing and cooling to 95-104 °F (35-40 °C).
- [0142] Mix vigorously for a minimum of 45 minutes or until all of the lumps have been removed and the batch is smooth and uniform. The batch may be recirculated through an 80 Mesh Filter throughout this Final Mix to help break down the lumps. Although shear (increased mixing and/or recirculating) is needed to break down the lumps, minimize aeration as much as possible (turbine mixer at vigorous counterclockwise direction; side sweep mixer at moderate clockwise). Mix for a minimum of 45 minutes.
- [0143] When the batch temperature is 95-104 °F (35-40 °C), analyze sample(s). Discontinue mixing and cooling.
- [0144] Adjustments that may be made are the following. If the pH is low, add sodium

hydroxide (20%) at a maximum of 5% of the total batch requirement. Remix and resample. If the pH is high, add aspartic acid at a maximum of 7.5% of the total batch requirement. Remix and resample. If the viscosity is low, mix additional 60 minutes and resample. Batch may also be recirculated through an 80 Mesh Filter.

[0145] Pumps that should be used for pumping material into the tanks include air diaphragm and positive displacement. Filters that should be used include 80 mesh for all transfer operations. The fill temperature should be 90-99 °F (32-37 °C). Do not reheat batch if product temperature falls below 90 °F (32 °C).

#### **EXAMPLE 2**

- [0146] This example demonstrates production of hair care compositions comprising a polyelectrolyte complex using a one-tank process.
- [0147] The process of Example 1 describes a two-tank process, where the polyelectrolyte complex is prepared in one tank, the remaining components are mixed in a separate tank, and the polyelectrolyte complex and the remaining components are combined. To simplify the process to one tank, the process is performed as described in Example 1, except the polyelectrolyte complex is first prepared in the tank and the additional components are added directly to the same tank after the polyelectrolyte complex forms. The final viscosity of the composition may be lower than if prepared in two tanks. To compensate, add additional Salcare® SC 96.

#### **EXAMPLE 3**

- [0148] This example demonstrates the compatibility of polyelectrolyte complexes (PECs) with certain charged compounds and polymers.
- [0149] The milky opaque appearance of 1 wt. % and 2 wt. % PVMMA/polyquat-28 PECs disappears in the presence of greater than 0.5 wt. % polyquaternium-4 (Celquat<sup>®</sup> L-200; Akzo Nobel, Amsterdam, Netherlands). This indicates that the structure of the PECs has been disrupted. Below 0.5 wt. % Celquat<sup>®</sup> L-200, the PECs are not disrupted. Celquat<sup>®</sup> H-100 at 0.5 wt. % did not disrupt the PECs. Celquat<sup>®</sup> L-200 and H-100 are similar, but L-200 has lower viscosity and higher cationic activity.
- [0150] Tinovis GTC/Acrylates/Beheneth-25 Methacrylate Copolymer et.al, 2.30%, product viscosity was water thin. Assumed to be rendered ineffective by PECs.
- [0151] Table 3 shows charged compounds and polymers that were combined with PECs.

These compositions showed no deleterious effects of split-end mending.

#### [**0152**] Table 3

Compound/Polymer	Amount (wt. %)
Stearamidopropyl	
Dimethylamine	2
(neutralized with Aspartic Acid)	
Bis(C13-15 Alkoxy) PG-	1
Amodimethicone	1
Aminopropyl Dimethicone	0.5
Polyquaternium-37	0.65-3.25
Polyquaternium-7	0.085
Polyquaternium-39	0.0425
DC 5-7113	
(Silicone Quaternium-16 (and)	
undeceth-11 (and) butyloctanol	1.0
(and) undeceth-5;	1.0
Dow Corning, Midland, MI,	
USA)	
Abil <sup>®</sup> T Quat 60	
(Silicone Quaternium-22;	1.0
Evonik, Essen, Germany)	
Quaternium-80	0.5
Merquat® 2001	
(Polyquaternium-47;	0.5
Nalco, Naperville, IL, USA)	
Vinyl	
Caprolactum/VP/Dimethyl-	
aminoethyl Methacrylate	0.25
Copolymer (in acid	
environment)	
Acrylic Acid/VP Crosspolymer	1.0

#### **EXAMPLE 4**

[0153] This example illustrates formulations comprising polyelectrolyte complexes and charged polymers.

[0154] All Compositions 4B-4E exhibited freeze-thaw stability. The present inventors also surprisingly found that Composition 4D, which contains Salcare<sup>®</sup> SC 96, exhibited superior long-term controlled viscosity (wherein the viscosity remained stable long-term), relative to Compositions 4B, 4C, and 4E.

[0155] Table 4 lists the ingredients of the hair care compositions. Comparative Composition 4A does not include PECs, whereas Compositions 4B-4E comprise PVMMA:polyquat-28 PECs and charged compounds and/or polymers. All compositions are

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rinse-out conditioners. The sodium hydroxide/citric acid/aspartic acid are adjusted to achieve the proper pH, if necessary.

[0156] Table 4

Ingredients	Comparative Composition	Composition 4B	Composition 4C	Composition 4D	Composition 4E
	4A	(wt. %)	(wt. %)	(wt. %)	(wt. %)
	(wt. %)	(*** 70)	(116.70)	(112.70)	(112.70)
DI Water	82.805	67.55	67.95	62.95	65.97
Citric Acid	0.1	0	0	0	0
L-Aspartic Acid	0.6	0.75	0.75	0.75	0.75
Stearamidopropyl	2	2	2	2	2
Dimethylamine					
Cetyl Alcohol	2.714	7	5	4	4
Stearyl Alcohol	5.286	0	0	1	0
Glyceryl Stearate	0	1.4	1	1	0.8
Isostearamidopropyl	0.145	0	0	0	0
Ethyldimonium					
Ethosulfate, PEG-9					
Crodamol STS	0	0	0	2	0
Montanav 202	0	0	0	0	1.18
Propylene Glycol	0	10	10	10	10
PVMMA (Gantrez®	0	0.2	0.2	0.2	0.2
S-97 BF)					
NaOH (20% active)	0	0.25	0.25	0.25	0.25
Polyquaternium-28	0	9	9	9	9
(Conditioneze® NT-					
20, 20% actives)					
Abil® T Quat 60	0	1	0	0	0
Quaternium-80,	0	0	1	0	0
Propylene Glycol					
Quat-18, Water	0.5	0	0	0	0
Salcare® SC 96	0	0	0	1	0
Merquat® 2001	0	0	0	0	0.5
Dimethicone	0	0	1	0	1
DC-8500	0	0	1	0	1
D5, D6,	0	0	0	5	0
Dimethicone					
Aminopropyl	0	0	0	0	0.5
Dimethicone					
D5, Dimethiconol	0	0	0	0	2
Cyclopentasiloxane,	5	0	0	0	0
Cyclohexasiloxane,					
Dimethicone					
Disodium EDTA	0.05	0.05	0.05	0.05	0.05
DMDM Hydantoin	0.2	0.2	0.2	0.2	0.2
Fragrance	0.6	0.6	0.6	0.6	0.6

**Target Specifications 4A:** 

pH: 4.00-5.00

Viscosity (RV T-B, 10 rpm, 1 min, 25C):

bulk: 5,000-9,000 cps

w/ shear (finished goods): 10,000-30,000 cps

Color: White (MS)

Odor: MS

Appearance: Viscous, opaque emulsion

**Target Specifications 4B:** 

pH: 4.50-6.00

Viscosity (RV T-B, 10 rpm, 1 min, 25C):

bulk: 7,000-15,000 cps

w/ shear (finished goods): 20,000-35,000 cps

Color: White (MS)

Odor: MS

Appearance: Viscous, opaque emulsion

**Target Specifications 4C:** 

pH: 4.50-6.00

Viscosity (RV T-B, 10 rpm, 1 min, 25C):

bulk: 7,000-15,000 cps

w/ shear (finished goods): 20,000-35,000 cps

Color: White (MS)

Odor: MS

Appearance: Viscous, opaque emulsion

**Target Specifications 4D:** 

pH: 4.50-6.00

Viscosity (RV T-B, 10 rpm, 1 min, 25C):

bulk: 7,000-15,000 cps

w/ shear (finished goods): 20,000-35,000 cps

Color: White (MS)

Odor: MS

Appearance: Viscous, opaque emulsion

**Target Specifications 4E:** 

pH: 4.50-6.00

Viscosity (RV T-B, 10 rpm, 1 min, 25C):

bulk: 7,000-15,000 cps

w/ shear (finished goods): 20,000-35,000 cps

Color: White (MS)

Odor: MS

Appearance: Viscous, opaque emulsion

#### **EXAMPLE 5**

[0157] This example demonstrates methods of making the compositions of Example 4.

[0158] For Comparative Composition 4A, start heating container to 80-85 °C and add the following: DI water, citric acid, and disodium EDTA. Ensure that the temperature is 80-85 °C and add the following: Quat-18/water, stearamidopropyl dimethylamine, cetyl alcohol, and stearyl alcohol. Vigorously mix for 30 minutes at 80-85 °C. Cool the mixture. At 57-60 °C, slowly add the L-aspartic acid. At 45 °C or below, add the following: isostearamidopropyl ethyldimonium ethosulfate/PEG-9,

cyclopentasiloxane/cyclohexasiloxane/dimethicone, fragrance, and DMDM hydantoin.

[0159] Compositions 4B, 4C, and 4E are prepared using two phases. For Composition 4B, in the Main Phase, add 52.05 wt. % (of the final total wt. % of the composition) of DI water. Start heating to 80-85 °C. Add the following one at a time and mix until fully dissolved and solution becomes clear: disodium EDTA and L-aspartic acid. Add the following one at a time at 80-85 °C and mix until melted: stearamidopropyl dimethylamine (check to make sure solution has turned clear), cetyl alcohol, and glyceryl stearate. Vigorously mix for 30 minutes at 80-85 °C. After 30 minutes, turn off heat, slow the mixing speed, and allow the composition to slowly cool. In the Side Phase, add 15.5 wt. % (of the final total wt. % of the composition) of DI water and slowly add the following, and mix until hydrated: PVMMA Copolymer (Gantrez® S-97 BF; ISP Corp., Wayne, NJ, USA) and sodium hydroxide (20% active). The solution will slightly thicken. The in-process pH should be 6.90-7.00. Heat the Side Phase to 50-55 °C. Slowly add the Polyquaternium-28 (Conditioneze® NT-20; ISP Corp.) under very vigorous mixing. The solution will turn opaque and become less viscous. Cool the Side Phase, and at ≤ 45 °C add 0.05 wt. % (of the final total wt. % of the composition) of DMDM hydantoin. Slowly add the Side Phase to the Main Phase at  $\leq 45$  °C. Then add the following: Abil<sup>®</sup> T Quat 60 (silicone quaternium-22; Evonik, Essen, Germany), propylene glycol, fragrance, and 0.15 wt. % (of the final total wt. % of the composition) of DMDM hydantoin.

[0160] For Composition 4C, in the Main Phase, add 52.45 wt. % (of the final total wt. % of the composition) of DI water. Start heating to 80-85 °C. Add the following one at a time and mix until fully dissolved and solution becomes clear: disodium EDTA and L-aspartic acid. Add the following one at a time at 80-85 °C and mix until melted: stearamidopropyl dimethylamine (check to make sure solution has turned clear), cetyl alcohol, and glyceryl

stearate. Vigorously mix for 30 minutes at 80-85 °C. After 30 minutes, turn off heat, slow the mixing speed, and allow the composition to slowly cool. In the Side Phase, add 15.5 wt. % (of the final total wt. % of the composition) of DI water and slowly add the following, and mix until hydrated: PVMMA Copolymer (Gantrez® S-97 BF; ISP Corp., Wayne, NJ, USA) and sodium hydroxide (20% active). The solution will slightly thicken. The in-process pH should be 6.90-7.00. Heat the Side Phase to 50-55 °C. Slowly add the Polyquaternium-28 (Conditioneze® NT-20; ISP Corp.) under very vigorous mixing. The solution will turn opaque and become less viscous. Cool the Side Phase, and at ≤ 45 °C add 0.05 wt. % (of the final total wt. % of the composition) of DMDM hydantoin. Slowly add the Side Phase to the Main Phase when at  $\leq$  45 °C. Pre-mix the quaternium-80/propylene glycol and dimethicone and then add to the combined Main and Side Phase. Then add the following: DC-8500 (Bis (C13-15 Alkoxy) PG Amodimethicone; Dow Corning, Midland, MI, USA), propylene glycol, fragrance, and 0.15 wt. % (of the final total wt. % of the composition) of DMDM hydantoin. For Composition 4D, add 40 wt. % (of the final total wt. % of the composition) of [0161] DI water. Slowly add the following, and mix until hydrated: PVMMA Copolymer (Gantrez® S-97 BF (ISP)) and sodium hydroxide (20% active). The solution will slightly thicken. The in-process pH should be 6.90-7.00. Heat to 50-55 °C, and slowly add the Polyquaternium-28 (Conditioneze® NT-20 (ISP)) under very vigorous mixing. The solution will turn opaque and become less viscous. Start heating to 80-85 °C, and add the following one at a time, mixing until fully dissolved: disodium EDTA, L-aspartic acid, and 22.95 wt. % (of the final total wt. % of the composition) of DI water. Add the following one at a time at 80-85 °C, and mix until melted: stearamidopropyl dimethylamine, cetyl alcohol, stearyl alcohol, glyceryl stearate, Crodamol STS (PPG-3 Benzyl Ether Myristate; Croda Inc., Edison, NJ, USA), Salcare SC 96 (65% polyquaternium-37, 25% propylene glycol dicaprylate/dicaprate, 10% PPG-1 trideceth-6; Ciba®/BASF, Basel, Switzerland). Mix vigorously for 30 minutes at 80-85 °C. Reduce the mixing speed and allow the composition to cool. When the temperature is <45 °C, add the following: D5/D6/Dimethicone, propylene glycol, fragrance, and DMDM hydantoin.

[0162] For Composition 4E, in the Main Phase, add 50.47 wt. % (of the final total wt. % of the composition) of DI water. Start heating to 80-85 °C. Add the following one at a time and mix until fully dissolved and solution becomes clear: disodium EDTA and L-aspartic acid. Add the following one at a time at 80-85 °C and mix until melted: stearamidopropyl dimethylamine (check to make sure solution has turned clear), cetyl alcohol, glyceryl

stearate, Montanov<sup>®</sup> 202 (arachidyl alcohol and behenyl alcohol and arachidyl glucoside; SEPPIC, Paris, France), and aminopropyl dimethicone. Vigorously mix for 30 minutes at 80-85 °C. After 30 minutes, turn off heat, slow the mixing speed, and allow the composition to slowly cool. In the Side Phase, add 15.5 wt. % (of the final total wt. % of the composition) of DI water and slowly add the following, and mix until hydrated: PVMMA Copolymer (Gantrez® S-97 BF; ISP Corp., Wayne, NJ, USA) and sodium hydroxide (20% active). The solution will slightly thicken. The in-process pH should be 6.90-7.00. Heat the Side Phase to 50-55 °C. Slowly add the Polyquaternium-28 (Conditioneze® NT-20; ISP Corp.) under very vigorous mixing. The solution will turn opaque and become less viscous. Cool the Side Phase, and at  $\leq 45$  °C add 0.05 wt. % (of the final total wt. % of the composition) of DMDM hydantoin. Slowly add the Side Phase to the Main Phase when at  $\leq$  45 °C. At 45 °C or below, add the Merquat<sup>®</sup> 2001 (polyquaternium-47; Nalco, Naperville, IL, USA). Pre-mix the D5/dimethiconol and dimethicone and then add to the combined Main and Side Phase. Then add the following: DC-8500 (Bis (C13-15 Alkoxy) PG Amodimethicone; Dow Corning, Midland, MI, USA), propylene glycol, fragrance, and 0.15 wt. % (of the final total wt. % of the composition) of DMDM hydantoin.

#### **EXAMPLE 6**

[0163] This example demonstrates the mending of split-ends of hair fibers using a test method of the present invention.

[0164] Table 5 compares the compositions used in this study that are presented in Figure 1. The sodium hydroxide/citric acid/aspartic acid are adjusted to achieve the proper pH, if necessary.

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[**0165**] Table 5

Ingredients	Composition	Composition	Composition 6C	Composition 6D
	6A	6B		1
	(wt. %)	(wt. %)	(wt. %)	(wt. %)
DI Water	69.95	67.95	62.95	65.97
L-Aspartic Acid	0.75	0.75	0.75	0.75
Stearamidopropyl	2	2	2	2
Dimethylamine				
Cetyl Alcohol	5	5	4	4
Stearyl Alcohol	0	0	1	0
Glyceryl Stearate	1	1	1	0.8
Crodamol STS	0	0	2	0
Montanav 202	0	. 0	0	1.18
Propylene Glycol	10	10	10	10
PVMMA (Gantrez®	0.2	0.2	0.2	0.2
S-97 BF)				
NaOH (20% active)	0.25	0.25	0.25	0.25
Polyquaternium-28	9	9	9	9
(Conditioneze® NT-				
$\hat{2}0$				
Abil® T Quat 60	1	0	0	0
Quaternium-80,	0	1	0	0
Propylene Glycol				
Salcare® SC 96	0	0	1	0
Merquat® 2001	0	0	0	0.5
Dimethicone	0	1	0	1
DC-8500	0	1	0	1
D5, D6,	0	0	5	0
Dimethicone				
Aminopropyl	0	0	0	0.5
Dimethicone				
D5, Dimethiconol	0	0	0	2
Disodium EDTA	0.05	0.05	0.05	0.05
DMDM Hydantoin	0.2	0.2	0.2	0.2
Fragrance	0.6	0.6	0.6	0.6

**[0166]** Table 6 compares compositions used in this study as presented in Figure 2. The sodium hydroxide/citric acid/aspartic acid are adjusted to achieve the proper pH, if necessary.

[**0167**] Table 6

Ingredients	Compo-	Compo-	Compo-	Compo-	Compo-	Compo-
	sition 6E	sition 6F	sition 6G	sition 6H	sition 6I	sition 6J
	(wt. %)	(wt. %				
						finished
						product)
DI Water	79.6	76.35	68.3	60.82	80.54	75.0752
L-Aspartic Acid	0	0.75	0.75	0.75	0	0
Citric Acid	0	0	0	0	0	0.0184
Stearamidopropyl	0	2	2	2	0	0
Dimethylamine						
Cocamidopropyl	0	0	0	0	0	0.368
betaine, water,						
sodium chloride						
Cetyl Alcohol	0	1	1	4	0	0
Stearyl Alcohol	0	0	0	0	0	0
Glyceryl Stearate	0	0.2	0.2	0.8	0	0
Crodamol STS	0	0	0	2	0	0
Aminomethyl	0	0	0	0	0.46	0
Propanol (AMP-95)						
PEG-40	0	0	0	0	0.6	0
hydrogenated castor						
oil						
Montanav 202	0	0	0	1.18	0	0
Glycerin	1	1	0	0	0	0
Sorbitol (70%)	0	0	0	0	0.5	0
Propylene Glycol	0.5	0.5	10	10	1	2.07
						(USP)
Polysorbate 20,	0	0	0	0	0	0.644
water						
PVMMA (Gantrez®	0.2	0.2	0.2	0.2	0.2	0.2
S-97 BF)						
NaOH (20% active)	0.25	0.25	0.25	0.25	0.25	0.25
Polyquaternium-28	9	9	9	9	9	9
(Conditioneze® NT-						
20, 20% active)						
Salcare® SC 96	3	3	3	1	0	0
Polyquaternium-4	0	0	0	0	0.5	0
(Celquat® H-100)						
Polyquaternium-4	0	0	0	0	0	0.46
(Celquat® L-200)						
PVP K-90 (20%)	0	0	0	0	1	0
Luviset® Clear	0	0	0	0	3.5	0
Polyquaternium-39	0	0	0	0	0.5	0
Ultrathix <sup>TM</sup> P-100	0	0	0	0	1	0
Advantage® S	0	0	0	0	0	0.23
Polyquaternium-11,	0	0	0	0	0	1.4444
water	_		_			
Polyquaternium-7	0	0	0	0	0	0.92
Dimethicone	3	2	1	1.5	0	0
	(DC-200,	(60,000	_			
	20 cst)	cst)				

Cyclopentasiloxane,	1	0	0	0	0	0
dimethiconol						
DC 2-8566	0	0	0	2	0	0
(Amodimethicone)						
DC-8500	0	0	1	0	0	0
DC-190	0	0	0	0	0.1	0
Aminopropyl	0	0	0.5	0.75	0	0
Dimethicone						
Phenyl trimethicone	2	2	0	0	0	0
(DC-556)						
D5, Dimethiconol	0	1	2	3	0	0
(DC-1501)						
DC 5-7113 silicone	0	0	0	0	0	0.92
quat microemulsion						
Disodium EDTA	0.05	0.05	0.05	0.05	0.05	0
DMDM Hydantoin	0.2	0.2	0.25	0.2	0.2	0
Fragrance	0.2	0.5	0.5	0.5	0.6	0.4
Isobutene and	0	0	0	0	0	8
propane and 152A						

**[0168]** Figure 1 shows the results of mending for conditioners when using twice-bleached European hair with split-ends produced mechanically. The results show about 80% mending with the rinse-off conditioners in three cycles.

**[0169]** Figure 2 shows the results of various compositions when using twice-bleached European hair with split-ends produced mechanically. The results are based on the assumption that all formulas are left in, including Night Repair. The results show greater than 80% for all leave-in compositions in one treatment.

#### EXAMPLE 7

[0170] This example demonstrates the mending of split-ends of hair fibers using a test method of the present invention.

[0171] Table 7 compares compositions used in this study. The sodium hydroxide/citric acid/aspartic acid are adjusted to achieve the proper pH, if necessary.

[**0172**] Table 7

Ingredients	Composition	Composition	Composition	Composition
	7A	7B	7C	7D
	(wt. %)	(wt. %)	(wt. %)	(wt. %)
DI Water	76.3497	41.2837	62.9497	68.3497
Disodium EDTA	0.05	0.1	0.05	0.05
L-Aspartic Acid	0.75	0	0.75	0.75
Stearamidopropyl	2	0	2	2
Dimethylamine				
Cetyl Alcohol	1	0	4	1

Stearyl Alcohol	0	0	1	0
Glyceryl Stearate	0.2	0	1	0.2
Crodamol STS	0	0	2	0
Cocamidopropyl	0	8.2	0	0
betaine, water, sodium				
chloride				
PVM/MA Copolymer	0.2	0.001	0.2	0.2
(Gantrez S-97 BF				
(ISP))				
Sodium Hydroxide	0.25	0.15	0.25	0.25
(20% active)				
Polyquaternium-28	9	0.045	9	9
(Conditioneze NT-20				
(ISP), 20% active)				
Dimethicone (60,000	2	0	0	1
cst)				
Phenyl Trimethicone	2	0	0	0
(DC-556)				
D5, Dimethiconol	1	0	0	0
(DC-1501)				
Fluid Blend	0	0	5	0
DC-8500	0	0	0	1
Aminopropyl	0	0	0	0.5
dimethicone		,		
Abil OSW 5	0	0	0	2
Glycerin	1	0	0	0
Propylene Glycol	0.5	0	10	10
Fragrance	0.5	0.7	0.6	0.5
DMDM Hydantoin	0.2	0	0.2	0.2
Cocos Nucifera	0.0001	0.0001	0.0001	0.0001
(Coconut) Oil				
Keratin Amino Acids	0.0001	0.0001	0.0001	0.0001
Flower/Leaf Extract	0.0001	0.0001	0.0001	0.0001
Salcare SC 96	3	0	1	3
Polyquaternium-10	0	0.8	0	0
Sodium laureth	0	32	0	0
sulfate-1 mole (25%)				
Disodium laureth	0	12.8	0	0
sulfosuccinate (about				
34% active)				
Styrene/VP	0	0.75	0	0
copolymer (40%),				
water				
PEG-6 caprylic/capric	0	0.6	0	0
triglyceride				
Kathon CG	0	0.07	0	0
Sodium chloride	0	2.5	0	0

#### **Target Specifications 7A:**

pH: 4.50-6.00

Viscosity (RV T-B, 10 rpm, 1 min, 25C): 5,000-15,000 cps

Color: White (MS)

Odor: MS

Appearance: Opaque emulsion

#### **Target Specifications 7B:**

pH: 5.5-6.5

Viscosity (LVT/spindle 4/30 rpm/ 1 min, 25C): 15,000-20,000 cps

Color: White/Off white (MS)

Odor: MS

Appearance: Viscous, opaque liquid

#### **Target Specifications 7C:**

pH: 4.50-6.00

Viscosity (RV T-B, 10 rpm, 1 min, 25C): 15,000-25,000 cps

Color: White (MS)

Odor: MS

Appearance: Viscous, opaque emulsion

#### **Target Specifications 7D:**

pH: 4.50-6.00

Viscosity (RV T-B, 10 rpm, 1 min, 25C): 10,000-20,000 cps

Color: White (MS)

Odor: MS

Appearance: Opaque emulsion

[0173] Fifty hair fibers containing split-ends were removed from hair tresses. The hair fiber samples were placed on glass slides aligned along a strip of double stick tape for optical imaging. Each fiber selected with a split-end was individually photographed and the image was stored before product application, using a WILD stereomicroscope at a magnification of about 25X with transmitted light emanating. The fibers were also rotated prior to image capture to make sure that the split-ends were in view.

[0174] The product application protocol listed below was followed. In each case, the 50 selected fibers with split-ends were placed respectively in five different tresses and then the product applied. Optical images of tip ends of the respective selected fibers were obtained again to examine and assess extent of split-end mending. The results appear in Table 8.

[0175] Table 8 shows a summary of the split-end data study comparing the total number of split-ends before treatment (50) to the number of split-ends repaired after treatment. (The

split-ends for the repeat study, category 6, were prepared by a separate method).

[0176] Table 8

Cate- gory	Composition	Initial Number Of Split- Ends	Number Mended	Number Partially Mended	Number Not Mended	Number Broke	% Mended (Mended + Partially Mended)
1	Leave in Treatment (Composition 7A)	50	32	13	3	2	94
2	Shampoo (Composition 7B)+Conditioner (Composition 7C)+Leave in Treatment (Composition 7A)	50	33	13	4	0	92
3	Overnight Treatment (Composition 7D)	50	30	16	3	1	94
4	Conditioner (Composition 7C)	49	39	3	7	0	86
5	Shampoo (Composition 7B)+Conditioner (Composition 7C)	50	29	4	14	3	70
6	Shampoo (Composition 7B)+Conditioner (Composition 7C) (Repeat) new stock	50	30	8	12	0	76

[0177] All categories show ability to mend split-ends in hair fibers, three of them showing improvements of 90% or more.

[0178] Product Application Protocol

[0179] Protocols are listed in the order presented in Table 8. To achieve 80% dry tresses for some compositions (as noted below), blow drying time was changed from 5 minutes to 20 minutes because of the nature of the blow dryer (mild air flow).

#### [0180] A. Pre-cleansing the Hair

- 1. Clean the wax-strip bound Platinum (2x) bleached hair by soaking in a 5% w/w solution of Sodium Lauryl Ether Sulfate (1M) solution. The solution to hair ratio is 40:1. Soak for 30 minutes at room temperature, and then massage the hair by hand for 3 minutes before rinsing under 40°C tap water until all the bubbles are rinsed out.
  - 2. Blot the hair dry with tissue paper or a towel, comb straight, and leave the hair

to dry at ambient room temperature overnight.

### [0181] B. Causing Split-Ends – Mechanically

- 1. The tresses are swatched by International Hair Importers in a way that the density of the hair is linear. Prepare 5 tresses that are 1.5" wide, cut and tape so that there is 6g of hair in each tress.
- 2. Attach each tress to the repeated combing machine and comb the tress 1500 times per side for a total of 3000 combings per tress.
- 3. During the combing, the blow dryer is positioned 8 inches from the tress and turned on high heat setting. The air flow is directed towards the bottom of the hair tress so that the hair is pushed towards the comb during repeated combing.
- [0182] C. Selecting the Split-Ends from the Tress and Taking Initial Pictures
- 1. Using a magnifying glass, examine the tip of the tresses to find hair fibers with split-ends. Select and remove 50 fibers with split-ends from the tress by the root. Select 10 split-end fibers out of each tress so that 50 fibers can be pulled from the five tresses.
- 2. Crimp the fibers at the root end using metal crimps, and then line up the crimps to be placed on tape so that there are 10 fibers on each tape strip. Cover up the crimps using another tape so that they can be easily picked up together.
- 3. Number all 50 fibers and take optical images of 10 out of the 50 hair fibers for before and after pictures.
- 4. Using a red permanent marker, mark each of the selected 50 fibers up to the beginning of the split.
- 5. Take optical images of 10 split-ends by mounting the split-ends on glass slides. A stereomicroscope at a magnification of 50X with transmitted light emanating from under the stage of the microscope is used to take the pictures.
- 6. Examine the other 40 fibers with split-ends using the stereomicroscope, but they do not need to be photographed.
- 7. The strip of tape that contains split-ends is placed back in the middle 1.5" tress.
- 8. Clamp the tress with the split-end fibers using a large clip such as a binder clip.
- [0183] D. Composition Application Category 1
  - 1. Wet the hair tress for 30 seconds under 35-40 °C water at 1 GPM.
  - 2. Apply 1.5 ml of a non-conditioning shampoo to the 1.5 inch tress and massage

for 1 minute.

- 3. Rinse the tress for 30 seconds under 35-40 °C water at 1 GPM.
- 4. Wring out the excess water by sliding the tress between fingers.
- 5. Mount the top of the tress to a stand for blow drying.
- 6. Clip the tress at <sup>3</sup>/<sub>4</sub> of its length with a hair clip to avoid flyway hair.
- 7. Blow dry the tress at medium heat with the dryer being 2 ft away from the tress for about 3 minutes.
- 8. Apply 1.5 ml of Composition 7A, leave in treatment, to the 1.5 inch damp tress evenly on both sides. Massage for 1 minute focusing to the ends and split-end prone areas.
  - 9. Comb once on each side of the tress.
  - 10. Mount the top of the tress to a stand for blow drying.
  - 11. Clip the tress at ¾ of its length with a hair clip to avoid flyway hair.
- 12. Blow dry the tress at medium heat (mild air flow) with the dryer being 2 ft away from the tress until it is 80% dry for about 20 minutes.
  - 13. After blow drying, comb the tress once on each side.
- [0184] D. Composition Application Category 2
  - 1. Wet the hair tress for 30 seconds under 35-40 °C water at 1 GPM.
- 2. Apply 1.5 ml of Composition 7B (shampoo) to the 1.5 inch tress and massage for 1 minute.
  - 3. Rinse the tress for 30 seconds under 35-40 °C water at 1 GPM.
  - 4. Wring out the excess water by sliding the tress between fingers.
- 5. Apply 1.5 ml of Composition 7C (conditioner) to the 1.5 inch tress and stroke the tress 9 times from top to bottom and let it stand for 1 minute.
  - 6. Rinse the tress for 30 seconds under 35-40 °C water at 1 GPM.
  - 7. Blot dry the tress gently using Kim Wipes.
  - 8. Mount the top of the tress to a stand for blow drying.
  - 9. Clip the tress at <sup>3</sup>/<sub>4</sub> of its length with a hair clip to avoid flyway hair.
- 10. Blow dry the tress at medium heat with the dryer being 2 ft away from the tress for 3 minutes.
- 11. Apply 1.5 ml of Composition 7A, leave in treatment, to the 1.5 inch damp tress. Massage for 1 minute focusing on the ends and split-end prone areas.
  - 12. Comb the tress once on each side.

- 13. Mount the top of the tress to a stand for blow drying.
- 14. Clip the tress at ¾ of its length with a hair clip to avoid flyway hair.
- 15. Blow dry the tress at medium heat (mild air flow) with the dryer being 2 ft away from the tress until it is 80% dry for about 20 minutes.
  - 16. After blow drying, comb through the tress twice on each side.
- [0185] D. Composition Application Category 3
  - 1. Wet the hair tress for 30 seconds under 35-40 °C water at 1 GPM.
- 2. Apply 1.5 ml of a non-conditioning shampoo to the 1.5 inch tress and massage for 1 minute.
  - 3. Rinse the tress for 30 seconds under 35-40 °C water at 1 GPM.
  - 4. Wring out the excess water by sliding the tress between fingers.
  - 5. Mount the top of the tress to a stand for blow drying.
  - 6. Clip the tress at <sup>3</sup>/<sub>4</sub> of its length with a hair clip to avoid flyway hair.
- 7. Blow dry the tress at medium heat with the dryer being 2 ft away from the tress for about 3 minutes.
- 8. Apply 1.5 ml of Composition 7D, overnight treatment, to the damp 1.5 inch tress to both sides of the tress. Massage for 1 minute focusing on the ends and split-end prone areas.
  - 9. Comb the tress once on each side.
- 10. Let the composition stand on the hair for 8 hours since this is an overnight treatment.
  - 11. Clip the tress at <sup>3</sup>/<sub>4</sub> of its length with a hair clip to avoid flyway hair.
- [0186] D. Composition Application Category 4
  - 1. Wet the hair tress for 30 seconds under 35-40 °C water at 1 GPM.
- 2. Apply 1.5 ml of a non-conditioning shampoo to the 1.5 inch tress and massage for 1 minute.
  - 3. Rinse the tress for 30 seconds under 35-40 °C water at 1 GPM.
  - 4. Wring out the excess water by sliding the tress between fingers.
- 5. Apply 1.5 ml of Composition 7C to the 1.5 inch tress and stroke the tress 9 times from top to bottom and let it stand for 1 minute.
  - 6. Rinse the tress for 30 seconds under 35-40 °C water at 1 GPM.
  - 7. Blot dry the tress gently using Kim Wipes.
  - 8. Mount the top of the tress to a stand for blow drying.

- 9. Clip the tress at <sup>3</sup>/<sub>4</sub> of its length with a hair clip to avoid flyway hair.
- 10. Blow dry the tress at medium heat with the dryer being 2 ft away from the tress until it is 80% dry for about 5 minutes.
  - 11. Comb through the tress twice on each side.
  - 12. Reapply the red markings on the fiber after blow drying
- 13. Repeat steps 1 to 12 for an additional 2 more cycles, so there is total of three treatments.
- [0187] D. Composition Application Category 5
  - 1. Wet the hair tress for 30 seconds under 35-40 °C water at 1 GPM.
- 2. Apply 1.5 ml of Composition 7B (shampoo) to the 1.5 inch tress and massage for 1 minute.
  - 3. Rinse the tress for 30 seconds under 35-40 °C water at 1 GPM.
  - 4. Wring out the excess water by sliding the tress between fingers.
- 5. Apply 1.5 ml of Composition 7C (conditioner) to the 1.5 inch tress and stroke the tress 9 times from top to bottom and let it stand for 1 minute.
  - 6. Rinse the tress for 30 seconds under 35-40 °C water at 1 GPM.
  - 7. Blot dry the tress gently using Kim Wipes.
  - 8. Comb through the tress once on each side.
  - 9. Mount the top of the tress to a stand for blow drying.
  - 10. Clip the tress at <sup>3</sup>/<sub>4</sub> of its length with a hair clip to avoid flyway hair.
- 11. Blow dry the tress at medium heat (mild air flow) with the dryer being 2 ft away from the tress until it is 80% dry for about 20 minutes.
  - 12. Comb through twice on each side.
- 13. Repeat steps 1 to 12 for an additional 2 more cycles, so there is a total of three treatments.
- [0188] E. Selecting the Split-ends from the Tress and Taking Final Pictures
- 1. Remove the strip of tape that contains split-ends from the middle of the 1.5" tress.
- 2. Using the stereomicroscope, take the after treatment optical images of the 10 hair fibers with split-ends by mounting the split-ends on glass slides.
  - 3. Examine the other 40 fibers with split-ends using the stereomicroscope.
  - 4. Count the number of fibers that have been mended to determine % mending.

#### **EXAMPLE 8**

[0189] This example demonstrates methods of communicating split-end mending to consumers.

**[0190]** Figure 3 is a schematic showing the mending of a split-end using polyelectrolyte complexes (PECs). The PECs are shown here in a possible mechanism of action where they attach to the site of the split-end, form a network with themselves and the fiber assembly, and shrink as they dry binding the split end from within.

[0191] Figures 4A-4D show still photography of the mending of a variety of split-end types using methods as described herein with compositions as described herein.

[0192] Figure 5 shows still frames from a video of the mending of split-ends through the process of mending method as described herein.

#### **EXAMPLE 9**

[0193] This example demonstrates additional compositions of the present invention.

### [0194] Composition 9A

Main Phase	wt%
DI Water	45.0397
Aminomethyl Propanol (AMP-95)	0.02
Add the following under very vigorous mixing for 30 mins.	
"Ultrathix P-100" (Acrylic Acid/VP Crosspolymer)	1
Add the following and mix until uniform	
Aminomethyl Propanol (AMP-95)	0.44
Side Phase 1	
DI Water	20
Sprinkle in the following and mix until clear	
Polyquaternium-4 (Celquat H-100)	0.5
Disodium EDTA	0.05
Liquid Additions:	
PVP K-90 (20% active)	1
VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active	3.5
Polyquaternium-39 (8.5% active)	0.5
Propylene Glycol	1
Sorbitol (70%)	0.5
Add Side Phase 1 to main tank and mix until uniform	
Side Phase 2	
DI Water	15.5
Slowly add the following, mix until hydrated	

PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.2
Sodium Hydroxide (20% active)	0.25
(solution will slightly thicken)	
In-process pH specification: 6.90-7.00	
Heat Side Phase to 50-55C	
Slowly add the following under very vigorous mixing	
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	9
(solution will turn opaque and become less viscous)	
DMDM Hydantoin	0.05
Slowly add Side Phase 2 to main tank and mix until	
uniform	
< 45C	
Pre-mix next two and fragrance	
PEG-40 Hydrogenated Castor Oil	0.6
PEG/PPG-18/18 Dimethicone (DC-190)	0.10
Fragrance	0.6
Cocos Nucifera (Coconut) Oil	0.0001
Keratin Amino Acids	0.0001
Flower/Leaf Extract	0.0001
DMDM Hydantoin	0.15

## **Target Specifications 9A:**

pH: 6.0-7.0

Viscosity (RV T-C, 5 rpm, 1 min,

25C): 40,000-80,000

Color: White (MS)

Odor: MS

Appearance: Viscous, opaque (hazy)

gel

## [0195] Composition 9B

Main Phase	wt%	wt%
DI Water	64.755325	59.5249
Add the following under vigorous mixing, mix until hydrated (clear)		
Polyquaternium-4 (Celquat L-200)	0.5	0.46
Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate Copolymer ("Advantage S" (ISP))	0.25	0.23
Citric Acid	0.02	0.0184
Liquid Additions:		
POLYQUATERNIUM-11, WATER (21.5% active)	1.57	1.4444
POLYQUATERNIUM-7 (8.5% active)	1	0.92

DC 5-7113 Silicone Quat Microemulsion - Dow Corning (66% Water, 25% Silicone Quaternium-16, 7.4% Undeceth-11, 4.5% Butyloctanol, 3.5% Undeceth-5)		
Butyloctanol, 3.3% Ondeceur-3)	1	0.92
PROPYLENE GLYCOL, USP	2.25	2.07
COCAMIDOPROPYL BETAINE (~30% active), WATER, SODIUM CHLORIDE	0.4	0.368
Side Phase		
DI Water	16.847826	15.55
Slowly add the following, mix until hydrated		
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.217391	0.2
Sodium Hydroxide (20% active)	0.271739	0.25
(solution will slightly thicken)		
In-process pH specification: 6.90-7.00		
Heat Side Phase to 50-55C		
Slowly add the following under very vigorous mixing		
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	9.782609	9
(solution will turn opaque and become less viscous)		
Slowly add side phase to main phase		
Pre-Mix polysorbate 20 and Fragrance		
POLYSORBATE 20	0.7	0.644
Fragrance	0.434783	0.4
Cocos Nucifera (Coconut) Oil	0.000109	0.0001
Keratin Amino Acids	0.000109	0.0001
Flower/Leaf Extract	0.000109	0.0001
Propellant Charge		
Isobutane and Propane and 152A (78.5% A46; 28.5% HFC 152A)	0	8

### **Target Specifications 9B:**

(Bulk) (Finished Goods)

**pH:** 5.0-6.5 5.0-6.5

**Color:** off white (MS) white (ms)

Odor: MS MS

Appearance: hazy liquid dense foam, non-watery, non-runny

Non-Volatiles:

Foam Stability: 200 seconds min.

**Vacuum:** 16-18 in.

Vapor Pressure @ 70F:69-74 psigVapor Pressure @ 130F:140-145 psigSpray Pattern/Diameter:Foam

# [0196] Composition 9C

Item	wt%
Main Phase	
DI Water	93.33163
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.001
NaOH, 20%	0.00125
Add the following and mix vigorously	
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	0.045
Disodium EDTA	0.02
PVP	0.45
Stearalkonium Chloride	1
Heat to 170-175F and mix for 30 min	
Cool the batch down to 90-95F	·
Glycerin	1
[DC-949]	
60% Amodimethicone, 5% Trideceth-12, 5% Cetrimonium	
Chloride, 30% Water	2
PEG-12 Dimethicone	0.5
Propylene Glycol	0.5
Triethanolamine	0.00001
Citric Acid	0.00001
DMDM Hydantoin	0.3
Hydrolyzed Silk	0.05
Water, Polysorbate20, Ascorbic Acid, Panthenol,	
Tocopherol Acetate, Biotin, Niacinamide	0.001
Water, Polyglyceryl-3 Distearate, Polysorbate 60, Myristic Acid, Palmitic Acid, Stearic Acid, Guar Hydroxypropyltrimonium Chloride, Triticum Vulgare (Wheat) Flour Lipids, Ceramide 3, Persea Gratissima (Avocado) Oil, Tetrahexyldecyl Ascorbate, Glycine Soja (Soybean) Sterols, Alpha-Glucan Oligosaccharide, Ethylhexyl Methoxycinnamate, Butyl Methoxydibenzoylmethane, Tocopheryl Acetate	0.0001
Premix the following and add to the batch	
Polysorbate20	0.4
Fragrance	0.4

### <u>Target</u> <u>Specification</u>

<u>9C:</u>

pH: 4.0-5.0

Color: Cloudy/Pale white

Odor: To match reference

Appearance: Milky liquid

# [0197] Composition 9D

Main Phase	wt%	wt%
DI Water	78.287935	72.0249
Add the following under vigorous mixing, mix until hydrated (clear)		
Polyquaternium-4 (Celquat H-100)	0.5	0.46
Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate Copolymer ("Advantage S" (ISP))	0.25	0.23
Citric Acid	0.02	0.0184
Liquid Additions:		
POLYQUATERNIUM-11, WATER (21.5% active)	1.57	1.4444
POLYQUATERNIUM-7 (8.5% active)	1	0.92
DC 5-7113 Silicone Quat Microemulsion - Dow Corning (66% Water, 25% Silicone Quaternium-16, 7.4% Undeceth-11, 4.5% Butyloctanol, 3.5% Undeceth-5)	1	0.92
PROPYLENE GLYCOL, USP	2.25	2.07
COCAMIDOPROPYL BETAINE (~30% active), WATER, SODIUM CHLORIDE	0.4	0.368
Side Phase		
DI Water	8.423913	7.75
Slowly add the following, mix until hydrated		
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.108696	0.1
Sodium Hydroxide (20% active)	0.13587	0.125
(solution will slightly thicken)		
In-process pH specification: 6.90-7.00		
Heat Side Phase to 50-55C		
Slowly add the following under very vigorous mixing		
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	4.891304	4.5
(solution will turn opaque and become less viscous)		
Add the following at $\leq 45$ C		
DMDM Hydantoin	0.027174	0.025
Slowly add side phase to main phase Pre-Mix polysorbate 20 and Fragrance		
POLYSORBATE 20	0.7	0.644
Fragrance	0.434783	0.4
Cocos Nucifera (Coconut) Oil	0.000109	0.0001
Keratin Amino Acids	0.000109	0.0001
Flower/Leaf Extract	0.000109	0.0001
Propellant Charge		
Isobutane and Propane and 152A (78.5% A46; 28.5% HFC 152A)	0	8

### **Target Specifications 9D:**

**Bulk** 

Color: off white (MS)

Odor: MS

Appearance: hazy liquid

5.0-6.5 pH:

Specific

Gravity:\* 0.986

Non-Volatiles: 4.4-4.9%

(T=4.66)

**Finished** 

**Product** 

Color: white (MS)

Odor: MS

Appearance: dense foam, non-

> nonwatery,

runny

pH: 5.0-6.5

Foam Stability: 200 seconds min

Foam Density:\*

0.039~g/mL

Specific

Gravity:\* 0.941

4.3-4.8%

Non-Volatiles: (T=4.58)

#### \* for reference

#### [0198] Composition 9E

Main Phase	wt%
DI Water	57.5647
Aminomethyl Propanol (AMP-95)	0.02
Add the following under very vigorous mixing for 30 mins.	
"Ultrathix P-100" (Acrylic Acid/VP Crosspolymer)	1
Add the following and mix until uniform	
Aminomethyl Propanol (AMP-95)	0.44
Side Phase 1	
DI Water	20
Sprinkle in the following and mix until clear	
Polyquaternium-4 (Celquat H-100)	0.5
Liquid Additions:	
PVP K-90 (20% active)	11
VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset	
Clear" (BASF)) - 20% active	3.5
Polyquaternium-39 (8.5% active)	0.5
Propylene Glycol	1

Sorbitol (70%)	0.5
Add Side Phase 1 to main tank and mix until uniform	
Side Phase 2	
DI Water	7.75
Slowly add the following, mix until hydrated	
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.1
Sodium Hydroxide (20% active)	0.125
(solution will slightly thicken)	
In-process pH specification: 6.90-7.00	
Heat Side Phase to 50-55C	
Slowly add the following under very vigorous mixing	
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	4.5
(solution will turn opaque and become less viscous)	
Add the following at $\leq 45$ C	
DMDM Hydantoin	0.025
Slowly add Side Phase 2 to main tank and mix until uniform	
< 45C	
Pre-mix next two and fragrance	
PEG-40 Hydrogenated Castor Oil	0.6
PEG/PPG-18/18 Dimethicone (DC-190)	0.1
Fragrance	0.6
Cocos Nucifera (Coconut) Oil	0.0001
Keratin Amino Acids	0.0001
Flower/Leaf Extract	0.0001
DMDM Hydantoin	0.175

## **Target Specifications 9E:**

pH: 6.0-7.0

Viscosity (RV T-C, 5 rpm, 1 min, 25C): 40,000-80,000

Color: White (MS)

Odor: MS

Appearance: Viscous, opaque (hazy)

gel

# [0199] Composition 9F

Item	wt%	wt%	wt%	wt%
Main Phase				
DI Water	74.2247	73.2247	71.2247	69.2247
Sprinkle in the following and mix until clear				
Polyquaternium-4 (Celquat H-100)	0.5	0.5	0.5	0.5
Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate Copolymer ("Advantage S" (ISP))	0.5	0.25	1	0.5

Liquid Additions				
POLYQUATERNIUM-11, WATER (20% active)	2.5	2.5	2.5	5
VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active	2.5	3.75	5	5
Polyquaternium-39 (8.5% active)	0.5	0.5	0.5	0.5
Propylene Glycol	1	1	1	1
Sorbitol (70%)	0.5	0.5	0.5	0.5
Side Phase				
DI Water	7.75	7.75	7.75	7.75
Slowly add the following, mix until hydrated				
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.1	0.1	0.10	0.1
Sodium Hydroxide (20% active)	0.125	0.125	0.125	0.125
(solution will slightly thicken)				
In-process pH specification: 6.90-7.00				
Heat Side Phase to 50-55C				
Slowly add the following under very vigorous mixing				
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	4.5	4.5	4.5	4.5
(solution will turn opaque and become less viscous)				
(mix for ~10 minutes)				
Add the following at ≤ 45C				
DMDM Hydantoin	0.025	0.025	0.025	0.025
Slowly add side phase to main phase at ≤ 45C				
< 45C				
Pre-mix next two and fragrance				
PEG-40 Hydrogenated Castor Oil	0.5	0.5	0.5	0.5
PEG/PPG-18/18 Dimethicone (DC-190)	0.1	0.1	0.1	0.1
Fragrance	0.5	0.5	0.5	0.5
DMDM Hydantoin	0.175	0.175	0.175	0.175
Cocos Nucifera (Coconut) Oil	0.0001	0.0001	0.0001	0.0001
Keratin Amino Acids	0.0001	0.0001	0.0001	0.0001
Flower/Leaf Extract	0.0001	0.0001	0.0001	0.0001
Add the following and mix until uniform				
"Sepigel 305"	4	4	4	4
Mix until uniform				

# **Target Specifications 9F:**

pH: 7.00-7.50

Viscosity (RV T-C, 5 rpm, 1 min, 25C): 50,000-100,000 cps

Color: clear-semi white

Odor: MS

Appearance: thick gel

[0200] Composition 9G

_				
Item	wt%	wt%	wt%	wt%
Main Phase	71.005	70.225	(0.225	66.205
DI Water	71.325	70.325	68.325	66.325
Sprinkle in the following and mix until clear	0.5	0.5	0.7	
Polyquaternium-4 (Celquat H-100)  Vinyl Caprolactum/VP/Dimethylaminoethyl	0.5	0.5	0.5	0.5
Methacrylate Copolymer ("Advantage S" (ISP))	0.5	0.25	1	0.5
Liquid Additions:				:
POLYQUATERNIUM-11, WATER (20% active)	2.5	2.5	2.5	5
VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active	2.5	3.75	5	5
Side Phase				
DI Water	7.75	7.75	7.75	7.75
Slowly add the following, mix until hydrated				
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.1	0.1	0.1	0.1
Sodium Hydroxide (20% active)	0.125	0.125	0.125	0.125
(solution will slightly thicken)				
In-process pH specification: 6.90-7.00				
Heat to 50-55C				
Slowly add the following under very vigorous mixing				
Polyquaternium-28 (Conditioneze NT-20 (ISP))	4.5	4.5	4.5	4.5
(solution will turn opaque and become less viscous)				
DMDM Hydantoin	0.025	0.025	0.025	0.025
"Salcare SC 96" - Ciba/BASF (65% Polyquaternium- 37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)	3	3	3	3
Mix until uniform				
<45C				
Silicone Premix				
Dimethicone (60,000 cst)	2	2	2	2
Phenyl Trimethicone (DC-556)	2	2	2	2
Cyclopentasiloxane, Dimethiconol	1	1	1	1
Glycerin	1	1	1	1
Propylene Glycol	0.5	0.5	0.5	0.5
Fragrance	0.5	0.5	0.5	0.5
DMDM Hydantoin	0.175	0.175	0.175	0.175

## **Target Specifications 9G:**

pH: 5.00-6.50

Viscosity (RV T-B, 10 rpm, 1 min, 25C): 20,000-40,000 cps

Color: Semi-Translucent - white (MS)

Odor: MS

Appearance: Viscous, opaque gel

## [0201] Composition 9H

Item	wt%	wt%
Main Phase		
DI Water	75.625	74.125
Sprinkle in the following and mix until clear		
Polyquaternium-4 (Celquat H-100)	0.5	0.5
Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate		
Copolymer ("Advantage S" (ISP))	0	0.25
POLYQUATERNIUM-11, WATER (20% active)	2.5	1.25
VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active	0	2.5
PEC Pre-mix		
DI Water	7.75	7.75
Slowly add the following, mix until hydrated		
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.1	0.1
Sodium Hydroxide (20% active)	0.125	0.125
(solution will slightly thicken)		
In-process pH specification: 6.90-7.00		
Heat to 50-55C		
Slowly add the following under very vigorous mixing		
Polyquaternium-28 (Conditioneze NT-20 (ISP))	4.5	4.5
(solution will turn opaque and become less viscous)		
mix for 10 minutes		
Add the following at $\leq$ 45C		
DMDM Hydantoin	0.025	0.025
Slowly add side phase to main phase at $\leq 45$ C		
< 45C		
Silicone Premix		
Dimethicone (60,000 cst)	2	2
Phenyl Trimethicone (DC-556)	2	2
Cyclopentasiloxane, Dimethiconol	1	1
Glycerin	1	1
Propylene Glycol	0.5	0.5
Fragrance	0.2	0.2
DMDM Hydantoin	0.175	0.175

"Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1		
Trideceth-6)	2	2
Mix until uniform		

## **Target Specifications 9H:**

pH: 5.00-6.50

Viscosity (RV T-B, 10 rpm, 1 min, 25C): 5,000-15,000 cps

Color: Semi-Translucent white (MS)

Odor: MS

Appearance: Viscous, milky liquid

## [0202] Composition 9I

Item	wt%	wt%	wt%
Main Phase			
DI Water	51.3247	62.3247	68.3247
Sprinkle in the following and mix until clear			
Polyquaternium-4 (Celquat H-100)	0.5	0.5	0.5
Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate			
Copolymer ("Advantage S" (ISP))	0.25	0.25	0.25
Disodium EDTA	0.05	0.05	0.05
L-Aspartic Acid	0.75	0.75	0.75
Add the following one at a time at 80-85C and mix until melted/homogeneous			
Stearamidopropyl Dimethylamine	2	2	2
Cetyl Alcohol	1	1	1
Glyceryl Stearate	0.2	0.2	0.2
Mix vigorously for 30 minutes @ 80-85C			
Reduce mixing speed; allow to cool and foam to reduce			
Liquid Additions:			
POLYQUATERNIUM-11, WATER (20% active)	1.25	1.25	1.25
VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active	2.5	2.5	2.5
Side Phase			
DI Water	7.75	7.75	7.75
Slowly add the following, mix until hydrated			
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.1	0.1	0.1
Sodium Hydroxide (20% active)	0.125	0.125	0.125
(solution will slightly thicken)			
In-process pH specification: 6.90-7.00			
Heat Side Phase to 50-55C			
Slowly add the following under very vigorous mixing			

Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	4.50	4.5	4.5
(solution will turn opaque and become less viscous)			
(mix for ~10 minutes)			
Add the following at ≤ 45C			
DMDM Hydantoin	0.025	0.025	0.025
Slowly add side phase to main phase at ≤ 45C			
< 45C			
"Abil OSW 5" - Evonik (84% Cyclopentasiloxane, 15% Dimethiconol, 1% Dimethicone)	20	10	5
"DC-8500" - Dow Corning (82% Bis(C13-15 Alkoxy) PG-Amodimethicone, 18% C14-15 Alcohols)	2	1.5	1
Phenyl Trimethicone (DC-556)	2	1.5	1
Fragrance	0.5	0.5	0.5
DMDM Hydantoin	0.175	0.175	0.175
Cocos Nucifera (Coconut) Oil	0.0001	0.0001	0.0001
Keratin Amino Acids	0.0001	0.0001	0.0001
Flower/Leaf Extract	0.0001	0.0001	0.0001
Add the following and mix until uniform			
"Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)	3	3	3
Mix until uniform			

## **Target Specifications 91:**

pH: 4.50-6.00

Viscosity (RV T-B, 10 rpm, 1 min, 25C): 20,000-30,000 cps

Color: White (MS)

Odor: MS

Appearance: Viscous opaque lotion

## [0203] Composition 9J

Item	wt%	wt%	wt%
Main Phase			
DI Water	25.8247	25.8247	25.8247
Glycerin	35	0	35
Propylene Glycol	0	35	0
Silicone Pre-Mix			
"Abil OSW 5" - Evonik (84% Cyclopentasiloxane, 15% Dimethiconol, 1% Dimethicone)	22	20	20
Amodimethicone (ADM 1100 (Wacker))	2	2	2
Phenyl Trimethicone (DC-556)	0	2	2
Fragrance	0.5	0.5	0.5
DMDM Hydantoin	0.175	0.175	0.175

Cocos Nucifera (Coconut) Oil	0.0001	0.0001	0.0001
Keratin Amino Acids	0.0001	0.0001	0.0001
Flower/Leaf Extract	0.0001	0.0001	0.0001
Sepigel 305	2	2	2
Mix moderately for ~30 minutes and until smooth			
Side Phase			
DI Water	7.75	7.75	7.75
Slowly add the following, mix until hydrated			
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.1	0.1	0.1
Sodium Hydroxide (20% active)	0.125	0.125	0.125
(solution will slightly thicken)			
In-process pH specification: 6.90-7.00			
Heat Side Phase to 50-55C			
Slowly add the following under very vigorous mixing			0.000
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	4.5	4.5	4.5
(solution will turn opaque and become less viscous)			
(mix for ~10 minutes)			
Add the following at ≤ 45C			**
DMDM Hydantoin	0.025	0.025	0.025
Slowly add side phase to main phase at ≤ 45C			
Mix until uniform			

## **Target Specifications 9J:**

pH: 7.0-8.0

Viscosity (RV T-B, 10 rpm, 1 min, 25C): 5,000-15,000 cps

Color: colorless-slightly yellow

Odor: MS

Appearance: slightly hazy oily liquid

## [0204] Composition 9K

Item	wt%	wt%	wt%
Main Phase			
DI Water	21.3247	31.3247	36.3247
Sprinkle in the following and mix until clear			
Polyquaternium-4 (Celquat H-100)	0.5	0.5	0.5
Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate			
Copolymer ("Advantage S" (ISP))	0.25	0.25	0.25
Glycerin	35	35	35
POLYQUATERNIUM-11, WATER (20% active)	1.25	1.25	1.25
VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear"		·	
(BASF)) - 20% active	2.5	2.5	2.5
Side Phase			

DI Water	7.75	7.75	7.75
Slowly add the following, mix until hydrated			
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.1	0.1	0.1
Sodium Hydroxide (20% active)	0.125	0.125	0.125
(solution will slightly thicken)			
In-process pH specification: 6.90-7.00			
Heat Side Phase to 50-55C			
Slowly add the following under very vigorous mixing			
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	4.5	4.5	4.5
(solution will turn opaque and become less viscous)			
(mix for ~10 minutes)			
Add the following at $\leq 45$ C			
DMDM Hydantoin	0.025	0.025	0.025
Slowly add side phase to main phase at $\leq 45$ C			
< 45C			
"Abil OSW 5" - Evonik (84% Cyclopentasiloxane, 15% Dimethiconol, 1% Dimethicone)	20	10	5
"DC-8500" - Dow Corning (82% Bis(C13-15 Alkoxy) PG-Amodimethicone, 18% C14-15 Alcohols)	2	1.5	1
Phenyl Trimethicone (DC-556)	2	1.5	. 1
Fragrance	0.5	0.5	0.5
DMDM Hydantoin	0.175	0.175	0.175
Cocos Nucifera (Coconut) Oil	0.0001	0.0001	0.0001
Keratin Amino Acids	0.0001	0.0001	0.0001
Flower/Leaf Extract	0.0001	0.0001	0.0001
Add the following and mix until uniform			
"Sepigel 305"	2	3	4

## **Target Specifications 9K:**

pH: 7.0-8.0

Viscosity (RV T-C, 10 rpm, 1 min, 25C): 50,000-100,000 cps

Color: clear-semi white

Odor: MS

Appearance: thick gel-thin liquid

## [0205] Composition 9L

Item	wt%
Main Phase	
DI Water	31.325
PEG-90M	0.5

77 00 070	
Heat to 90-95C	
(glycerin can be added while heating)	12
Glycerin	12
Side Phase 1	
Add the following in an appropriate side phase container, heat to 90-95C, and mix under very moderate mixing	
Mineral Oil (Paraffinum Liquidum)	9
Isoceteth-20	22
Oleth-2	8
After fully homogenous, add side phase to main phase	
Mix vigorously for 45 mins @ 90-95C, then allow to slowly cool	
Side Phase 2	
DI Water	7.75
Slowly add the following, mix until hydrated	
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.1
Sodium Hydroxide (20% active)	0.125
(solution will slightly thicken)	
In-process pH specification: 6.90-7.00	
Heat Side Phase to 50-55C	
Slowly add the following under very vigorous mixing	
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	4.5
(solution will turn opaque and become less viscous)	******
Add the following at $\leq 45$ C	
DMDM Hydantoin	0.025
Slowly add side phase to main phase @ 60-70C	
VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active	0.2
VP/VA Copolymer, Water	2.8
Methoxy PEG/PPG-7/3 Aminopropyl Dimethicone	1
Add the following at 45C	
DMDM Hydantoin	0.175
Fragrance	0.5

## **Target Specifications 9L:**

pH: 3.20-4.50

Penetration (50g weight w/ cone) 18-24mm

Color: Colorless

Odor: MS

Appearance: thick gel wax

## [0206] Composition 9M

Item	wt%
Main Phase	
DI Water	31.2837
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.001
NaOH, 20%	0.00125
Add the following and mix vigorously	
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	0.045
Slowly add the following, mix until hydrated	
Polyquaternium 10	0.8
Heat to 50C and mix until clear	
Disodium EDTA	0.1
COCAMIDOPROPYL BETAINE (~30% active),	
WATER, SODIUM CHLORIDE	8.2
NaOH, 20%	0.14875
Sodium Laureth Sulfate- 1 mole (25%)	32
Disodium Laureth Sulfosuccinate (~34% active)	12.8
Styrene/ VP Copolymer (40%), Water	0.75
PEG-6 Caprylic/Capric Triglyceride	0.6
Kathon CG	0.07
Fragrance	0.7
Cocos Nucifera (Coconut) Oil	0.0001
Keratin Amino Acids	0.0001
Flower/Leaf Extract	0.0001
Premix the following and add to the batch	
DI Water	10
Sodium Chloride	2.5

## **Target Specifications 9M:**

pH: 5.5-6.5

Viscosity (LVT/spindle 4/30 rpm/1 min, 25C): 15,000-20,000 cps

Color: White/Off white (MS)

Odor: MS

Appearance: Viscous, opaque liquid

# [0207] Composition 9N

Item	wt%	wt%
Main Phase		
DI Water	47.4497	60.2497
Start heat to 80-85C		
Add the following one at time, mix until fully dissolved.		
Disodium EDTA	0.05	0.05
L-Aspartic Acid	0.75	0.75
Add the following one at a time at 80-85C and mix until melted		
Stearamidopropyl Dimethylamine	2	2
Cetyl Alcohol	4	1
Stearyl Alcohol	1	0
Glyceryl Stearate	1	0.2
"Crodamol STS" (100% PPG-3 Benzyl Ether Myristate)	2	0
Mix vigorously for 30 minutes @ 80-85C		
Reduce mixing speed and allow to cool		
Add the following @ 65-70C		
Propylene Glycol	10	0
Side Phase		
DI Water	15.5	15.5
Slowly add the following, mix until hydrated		
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.2	0.2
Sodium Hydroxide (20% active)	0.25	0.25
(solution will slightly thicken)		
In-process pH specification: 6.90-7.00		
Heat Side Phase to 50-55C		
Slowly add the following under very vigorous mixing		
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	9	9
(solution will turn opaque and become less viscous)		
(mix for ~10 minutes)		
Add the following at $\leq 45$ C		
DMDM Hydantoin	0.05	0.05
Slowly add side phase to main phase at $\leq 45$ C		
< 45C		
Fluid Blend	5	5
Fragrance	0.6	0.6
Cocos Nucifera (Coconut) Oil	0.0001	0.0001
Keratin Amino Acids	0.0001	0.0001
Flower/Leaf Extract	0.0001	0.0001
DMDM Hydantoin	0.15	0.15
Add the following and mix until uniform		

"Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25%		
Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)	1	5

## **Target Specifications 9N:**

pH: 4.50-6.00

Viscosity (RV T-B, 10 rpm, 1 min, 25C): 15,000-25,000 cps

Color:

White (MS)

Odor: MS

Appearance: Viscous, opaque emulsion

#### Composition 9O [0208]

Item	wt%
Main Phase	
DI Water	60.8497
Disodium EDTA	0.05
L-Aspartic Acid	0.75
Add the following one at a time at 80-85C and mix until melted/homogeneous	
Stearamidopropyl Dimethylamine	2
Cetyl Alcohol	1
Glyceryl Stearate	0.2
Mix vigorously for 30 minutes @ 80-85C	
Reduce mixing speed; allow to cool and foam to reduce	
Side Phase	
DI Water	15.5
Slowly add the following, mix until hydrated	
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.2
Sodium Hydroxide (20% active)	0.25
(solution will slightly thicken)	
In-process pH specification: 6.90-7.00	
Heat Side Phase to 50-55C	
Slowly add the following under very vigorous mixing	
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	9
(solution will turn opaque and become less viscous)	
(mix for ~10 minutes)	
Add the following at ≤ 45C	
DMDM Hydantoin	0.05
Slowly add side phase to main phase at ≤ 45C	
< 45C	
Silicone Pre-Mix	
Dimethicone (60,000 cst)	2
Phenyl Trimethicone (DC-556)	2
D5, Dimethiconol (DC-1501)	1

,	
Glycerin	1
Propylene Glycol	0.5
Fragrance	0.5
DMDM Hydantoin	0.15
Cocos Nucifera (Coconut) Oil	0.0001
Keratin Amino Acids	0.0001
Flower/Leaf Extract	0.0001
Add the following and mix until uniform	
"Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25%	
Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-	
6)	3

## **Target Specifications 90:**

pH: 4.50-6.00

Viscosity (RV T-B, 10 rpm, 1 min, 25C): 5,000-15,000 cps

Color: White (MS)

Odor: MS

Appearance: opaque emulsion

## [0209] Composition 9P

Item	wt%
Main Phase	
DI Water	52.8497
Disodium EDTA	0.05
L-Aspartic Acid	0.75
Add the following one at a time at 80-85C and mix until melted/homogeneous	
Stearamidopropyl Dimethylamine	2
Cetyl Alcohol	1
Glyceryl Stearate	0.2
Aminopropyl Dimethicone	0.5
Mix vigorously for 30 minutes @ 80-85C	
Reduce mixing speed; allow to cool and foam to reduce	
Side Phase	
DI Water	15.5
Slowly add the following, mix until hydrated	
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.2
Sodium Hydroxide (20% active)	0.25
(solution will slightly thicken)	
In-process pH specification: 6.90-7.00	
Heat Side Phase to 50-55C	
Slowly add the following under very vigorous mixing	
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	9

(solution will turn opaque and become less viscous)	
(mix for ~10 minutes)	
Add the following at $\leq 45$ C	
DMDM Hydantoin	0.05
Slowly add side phase to main phase at ≤ 45C	
<45C	
Silicone Pre-Mix	
"Abil OSW 5" - Evonik (84% Cyclopentasiloxane, 15%	
Dimethiconol, 1% Dimethicone)	2
Dimethicone (60,000 cst)	1
"DC-8500" - Dow Corning (82% Bis(C13-15 Alkoxy) PG- Amodimethicone, 18% C14-15 Alcohols)	1
Propylene Glycol	10
Fragrance	0.5
DMDM Hydantoin	0.15
Cocos Nucifera (Coconut) Oil	0.0001
Keratin Amino Acids	0.0001
Flower/Leaf Extract	0.0001
Add the following and mix until uniform	l
"Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-	
6)	3

## **Target Specifications 9P:**

pH: 4.50-6.00

Viscosity (RV T-B, 10 rpm, 1 min, 25C): 10,000-20,000 cps

Color: White (MS)

Odor: MS

Appearance: opaque emulsion

## [0210] Composition 9Q

Item	wt%
Main Phase	
DI Water	52.8
Disodium EDTA	0.05
L-Aspartic Acid	0.75
Add the following one at a time at 80-85C and mix until melted/homogenous	
Stearamidopropyl Dimethylamine	2
Cetyl Alcohol	1
Glyceryl Stearate	0.2
Aminopropyl Dimethicone	0.5
Mix Vigorously for 30 minutes @ 80-85C	
Reduce mixing speed and allow to cool	

Side Phase	
DI Water	15.5
Slowly add the following, mix until hydrated	
PVM/MA Copolymer (Gantrez S-97 BS (ISP))	0.2
Sodium Hydroxide (20% Active)	0.25
(solution will slightly thicken)	
In-process pH specification: 6.90-7.00	
Heat side Phase to 50-55C	
Slowly add the following under very rigorous mixing	
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% Active)	9
(solution will turn opaque and become less viscous)	
Add the following at ≤ 45C	
DMDM Hydantoin	0.05
Slowly add side phase to main phase at ≤45C	
< 45C	
Pre-mix next two	
DS, Dimethiconol	2
Dimethicone	1
DC-8500	1_
Propylene Glycol	10
Fragrance	0.5
DMDM Hydantoin	0.2
"Salcare SC 96"	3

## Target Specifications 9Q:

pH:

4.50-6.00

Viscosity (RV T-8, 10 rpm, 1 min, 25C):

bulk:

7,000-15,0000 cps

Color:

White (MS)

Odor:

MS

Appearance:

opaque emulsion

#### [0211] Composition 9R

Item	wt%
Main Phase	
DI Water	40.55
Slowly add the following, mix until hydrated	
PVM/MA Copolymer (Gantrez S-97 BS (ISP))	0.2
Sodium Hydroxide (20% Active)	0.25
(solution will slightly thicken)	
In-process pH specification: 6.90-7.00	
Heat to 50-55C	

Slowly add the following under very rigorous mixing	
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% Active)	9
(solution will turn opaque and become less viscous)	
Start heat to 80-85C	
Add the following one at a time, mix until fully dissolved.	
Disodium EDTA	0.05
L-Aspartic Acid	0.75
DI Water	20.27
Add the following one at a time at 80-85C and mix until melted/homogenous	
Stearamidopropyl Dimethylamine	2
Cetyl Alcohol	4
"Montanov 202"	1.18
Glyceryl Stearate	0.80
"Crodamol STS" (Benzyl Ether Myristate)	2
"Salcare SC 96"	1
Aminopropyl Dimethicone	0.75
Mix vigorously for 30 minutes @ 80-85C	
Reduce mixing speed and allow to cool.	
< 45C	
Pre-mix next two	
DS, Dimethiconol	3
Dimethicone	1.5
DC 2-8566 (Amodimethicone)	2
Propylene Glycol	10
Fragrance	0.5
DMDM Hydantoin	0.2

## Target Specifications 9R:

pH: 4.50-6.00

Viscosity (RV T-8, 10 rpm, 1 min, 25C):

bulk:

15,0000-25,000 cps

White (MS) Color:

Odor: MS

opaque emulsion Appearance:

[0212] Composition 9S

Main Phase	wt%	wt%	wt%	wt%	wt%	wt%
DI Water	64.75565217	59.5252	64.86434783	59.6252	64.75565217	59.5252
Add the following		0310202				
under vigorous mixing,						
mix until hydrated						
(clear)	;					
Polyquaternium-4						
(Celquat L-200)	0.5	0.46	0.5	0.46	0.5	0.46
"Advantage S" (ISP)	0.25	0.23	0.25	0.23	0.25	0.23
Citric Acid	0.02	0.0184	0.02	0.0184	0.02	0.0184
Liquid Additions:						
POLYQUATERNIUM-						
11, WATER (AQUA)	1.57	1.4444	1.57	1.4444	1.57	1.4444
		0.92	1.57	0.92		0.92
POLYQUATERNIUM-7 DC S-7113 Silicone	1	0.92	<u> </u>	0.92	1	0.92
Quat Microemulsion						
(Dow Corning)	1	0.92	1	0.92	1	0.92
PROPYLENE	1	0.72	1	0.72	1	0.72
GLYCOL, USP	2.25	2.07	2.25	2.07	2.25	2.07
COCAMIDOPROPYL	2.23	2.07	2.23	2.07	2.23	٠٠٠/
BETAINE, WATER,						
SODIUM CHLORIDE	0.4	0.368	0.4	0.368	0.4	0.368
Side Phase						
40.0	16.84782609	15.55	16.84782609	15.55	16.84782609	15.55
DI Water	10.64762009	13.33	10.04/02009	13.33	10.64762009	13.33
Slowly add the following, mix until						
hydrated.						
PVM/MA Copolymer		-				
(Gantrez S-97 BS (ISP))	0.2173913	0.2	0.2173913	0.2	0.2173913	0.2
Sodium Hydroxide (20%	0.2173713	0.2	0.21,0313	0,2	0.21,0315	
Active)	0.27173913	0.25	0.27173913	0.25	0.27173913	0.25
(solution will slightly						
thicken)						
In-process pH						
specification: 6.90-7.00						
Heat to 50-55C						
Slowly add the						
following under very						
rigorous mixing						
Polyquaternium-28						
(Conditioneze NT-20						
(ISP))	9.7826087	9	9.7826087	9	9.7826087	9
(solution will turn						
opaque and become less						
viscous)						
Slowly add side phase						
to main phase.						
Pre-mix next and						
Fragrance				1		
POLYSORBATE 20,	0.70	0.644	0.70	0.644	0.7	0.644

WATER						
Fragrance	0.43478261	0.4	0.32608696	0.3	0.43478261	0.4
Propellant Charge						
Isobutane and Propane and 152A	0	8	0	8	0	8

## Target Tentative Specifications 9S (also for Composition 6J):

(Bulk)

(Finished Goods)

pH:

5.0-6.5

5.0-6.5

color:

off white (MS)

white (ms) MS

odor:

appearance:

MS hazy milky liquid

dense foam, non-watery, non-runny

## [0213] Composition 9T

Item	wt%
Main Phase	
DI Water	40.55
Slowly add the following, mix until hydrated	
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.2
Sodium Hydroxide (20% active)	0.25
(solution with slightly thicken)	
In-process pH specification: 6.90-7.00	
Record pH: 6.95	
Heat to 50-55C	
Slowly add the following under very vigorous mixing	
Polyquaternium-28 (Conditioneze NT-20 (ISP))	9
(solution will turn opaque and become less viscous)	
DI Water	39.05
Disodium EDTA	0.05
"Salcare SC 96"	3
Mix until uniform	
<45C	
Dimethicone (DC-200, 20 cst)	3
Phenyl Trimethicone (DC-556)	2
Cyclopentasiloxane, Dimethiconol	1
Glycerin	1
Propylene Glycol	0.5
Fragrance	0.2
DMDM Hydantoin	0.2

## <u>Target Specifications 9T:</u>

pH 5.00-6.50

pH Viscosity (RV T-B, 10 rpm, 1 min, 25C) Bulk:

Bulk: 7,000-15,000 cps

Color Semi-Translucent white (MS)

Odor MS

Appearance Viscous, milky fluid

## [0214] Composition 9U

Item	wt%
Main Phase	
DI Water	60.85
Disodium EDTA	0.05
L-Aspartic Acid	0.75
Add the following one at a time at 80-85C and mix until melted/homogenous	
Stearamidopropyl Dimethylamine	2
Cetyl Alcohol	1
Glycerol Stearate	0.2
Mix vigorously for 30 minutes @80-85C	
Reduce mixing speed; allow to cool and foam to reduce	
Side Phase	
DI Water	15.50
Slowly add the following; mix until hydrated	
PVM/MA Copolymer (Gantrez S-97)BF (ISP))	0.2
Sodium Hydroxide (20% active)	0.25
(solution will slightly thicken)	
In-process pH specification 6.90-7.00	
Heat Side Phase to 50-55C	
Slowly add the following under very vigorous mixing	
Polyquaternium-28 (Conditioneze NT-20 (ISP))	9
(solution will turn opaque and become less viscous)	
(mix for ~10 minutes)	
add the following at ≤45C	
DMDM Hydantoin	0.05
Slowly add side phase to main phase at ≤45C	
<45C	
Silicone Pre-Mix	
Dimethicone (60,000 cst)	2
Phenyl Trimethicone (Dc-556)	2
DS, Dimethiconol (DC-1501)	1
Glycerin	1
Propylene Glycol	0.5
Fragrance	0.5
DMDM Hydantoin	0.15

1			
١	"Salcare SC 96"	İ	3

## Target Specifications 9U:

pH 4.5-6.50

Viscosity (RV T-B, 10 rpm, 1 min, 25C)

Bulk: 8,000-16,000 cps

Color White (MS)

Odor MS

Appearance opaque emulsion

## [0215] Composition 9V

Item	wt%	wt%	wt%
Main Phase			
DI Water	24.99	24.99	24.99
Sprinkle in the following and mix until clear			
Polyquaternium-4 (Celquat H-100)	0.5	0.5	0.5
Disodium EDTA	0.05	0.05	0.05
Liquid Additions:			
PVP K-90 (20%)	1	1	1
"Luviset Clear" (BASF)	3.5	3.5	3.5
Polyquaternium 39	0.5	0.5	0.5
Propylene Glycol	1	1	1
Sorbitol 70%	0.5	0.5	0.5
Aminomethyl Propanol (AMP -95)	0.15	0.15	0.15
Side Phase 1			
DI Water	40	40	40
Aminomethyl Propanol (AMP-95)	0.02	0.02	0.02
Add the following under very vigorous mixing for 30 mins.			
"Ultrathix P-100" (Acrylic Acid/VP Crosspolymer)	1	1	1
Add the following and mix until uniform			
Aminomethyl Propanol (AMP-95)	0.15	0.15	0.15
Add Side Phase 1 to main tank and mix until uniform			
Add the following and mix until uniform			
Aminomethyl Propanol (AMP-95)	0.14	0.14	0.14
Side Phase 2			
DI Water	15.55	15.55	15.55
Slowly add the following; mix until hydrated			
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.2	0.2	0.2
Sodium Hydroxide (20% active)	0.25	0.25	0.25
(solution will slightly thicken)			
In-process pH specification: 6.90-7.00			
Heat Side Phase to 50-55C			
Slowly add the following under very vigorous mixing			
Polyquaternium-28 (Conditioneze NT-20 (ISP))	9	9	9

(solution will turn opaque and become less viscous)			
Slowly add Side Phase 2 to main tank			
<45C			
Pre-mix next two and fragrance			
PEG-40 Hydrogenated Castor Oil	0.6	0.6	0.6
DC-190	0.1	0.1	0.1
Fragrance	0.6	0.6	0.6
DMDM Hydantoin	0.2	0.2	0.2

Target Specifications 9V:

pH: 6.5-7.5

Viscosity (RV T-C, 5 rpm, 1 min, 25C):

bulk: 40,000-90,000

Color: White (MS)

Odor: MS

Appearance: Viscous, opaque gel

[0216] Composition 9W

Item	wt%	wt%	wt%
Main Phase			
DI Water	47.4497	60.2497	60.25
Start heat to 80-85C			
Add the following one at a time, mix until fully dissolved			
Disodium EDTA	0.05	0.05	0.05
L-Aspartic Acid	0.75	0.75	0.75
Add the following one at a time at 80-85C and mix until melted			
Stearamidopropyl Dimethylamine	2	2	2
Cetyl Alcohol	4	1	1
Stearyl Alcohol	1	0	0
Glyceryl Stearate	1	0.2	0.2
"Crodamol STS" (100% PPG-3 Benzyl Ether Myristate)	2	0	0
Mix Vigorously for 30 minutes @ 80-85C			
Reduce mixing speed and allow to cool			
Add the following at 65-70C			
Propylene Glycol	10	0	0
Side Phase			
DI Water	15.5	15.5	15.5
Slowly add the following, mix until hydrated			
PVM/MA Copolymer (Gantrez S-97 BS (ISP))	0.2	0.2	0.2
Sodium Hydroxide (20% Active)	0.25	0.25	0.25

(solution will slightly thicken)			
In-process pH specification: 6.90-7.00			
Heat side Phase to 50-55C			
Slowly add the following under very rigorous mixing			
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% Active)	9	9	9
(solution will turn opaque and become less viscous)			
(mix for "10 minutes)			
Add the following at $\leq 45$ C			
DMDM Hydantoin	0.05	0.05	0.05
Slowly add side phase to main phase at ≤43C			
< 45C			
Fluid Blend	5	5	5
Fragrance	0.6	0.6	0.6
Coca Nucifers (Coconut) Oil	0.0001	0.0001	0
Keratin Amino Acids	0.0001	0.0001	0
Flower/Leaf Extract	0.0001	0.0001	0
DMDM Hydantoin	0.15	0.15	0.15
Add the following and mix until uniform			
"Salcare SC 96"- Ciba/BASF (65% Polyquaternium-37,			
25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)	1	5	5

## Target Specifications 9W:

4.5-6.00

pH Viscosity (RV T-8, 10 rpm, 1 min, 25C) 15,000-25,000 cps

White (MS) Color

Odor

Appearance Viscous, opaque emulsion

#### Composition 9X [0217]

Item	wt%	wt%
Main Phase		
DI Water	54.45	52.05
Start heat to 80-85C		
Add the following one at a time, mix until fully dissolved and solution becomes clear		
Disodium EDTA-	0.05	0.05
L-Aspartic Acid	0.75	0.75
Add the following one at a time at 80-85C and mix until melted		
Stearamidopropyl Dimethylamine	2	2
Check to make sure solution has turned clear		

Cetyl Alcohol	5	7
Glyceryl Stearate	1	1.4
Vigorously mix for 30 minutes at 80-85C		
After 30 minutes, turn off heat, slow mixing speed and allow to slowly cool		
Side Phase		
DI Water	15.5	15.5
Slowly add the following, mix until hydrated		
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.2	0.2
Sodium Hydroxide (20% active)	0.25	0.25
(solution will slightly thicken)		
In-process pH specification: 6.90-7.00		
Heat Side Phase to 50-55C		
Slowly add the following under very vigorous mixing		
Polyquaternium-28 (Conditioneze NT-20 (ISP))	9	9
(solution will turn opaque and become less viscous)		
Add the following at ≤45C		
DMDM Hydantoin	0.05	0.05
Slowly add side phase to main phase at ≤45C		
Abil T Quat 60 (Evonik) – Silicone Quaternium – 22	1	1
Propylene Glycol	10	10
Fragrance	0.6	0.6
DMDM Hydantoin	0.15	0.15

### **Target Specifications 9X:**

pH: 4.50-6.00

Viscosity (RV T-B, 10 rpm, 1

min, 25C):

Bulk: 7,000-15,000 cps

w/ shear (finished goods): 20,000-35,000 cps

Color: White (MS)

Odor: MS

Appearance: Viscous, opaque emulsion

#### **EXAMPLE 10**

[0218] This example illustrates process parameters for forming PECs.

[0219] - Water Densities: Perry's Chemical Engineer's Handbook.

[0220] - densities for NaOH 20% = 1.2191 kg/L and Conditioneze = 1.042 kg/L.

### [**0221**] One 270-kg batch

the PEC Complex in 113-liter / 30-gallon premix vessel with center turbine agitator only main batch in 227-liter / 60-gallon mixing vessel with center turbine and side sweep agitators

Processing Parameters for PEC Complex:

### [0222] <u>Initial Water Charge</u> = 42.02 L (41.85 kg)

Addition Rate = 28 L/min (28 kg/min)

Total Addition Time = 1.5 minutes

Initial Water Temperature = 29°C

Center Turbine Agitator = Moderate (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

## [0223] $\underline{PVM/MA Copolymer (Gantrez S-97 BF)} = 0.54 \text{ kg (solid)}$

Addition Rate = 0.6 kg/min

Total Addition Time = 1 minute

Temperature =  $30^{\circ}$ C

Center Turbine Agitator = Moderate (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Dispersion Mix Time = 30 minutes

### [0224] Sodium Hydroxide, 20% = 0.55 L (0.675 kg)

Addition Rate = 0.6 L/min (0.7 kg/min)

Total Addition Time = 1 minute

Temperature =  $30^{\circ}$ C

Center Turbine Agitator = Slow (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Hydration Mix Time = 20 minutes

In-Process pH Check = 6.90

Heat Premix for 10 minutes to 51°C

### [0225] Polyquaternium 28 (Conditioneze NT-20) = 23.32 L (24.30 kg)

Addition Rate = 3.33 L/min (3.47 kg/min)

Total Addition Time = 7 minutes

Temperature =  $49^{\circ}$ C

Center Turbine Agitator = Moderate/High (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Complex Mix Time = 40 minutes

### [0226] Premix Cool Down

Cool to 29°C

Center Turbine Agitator = Very Slow (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Cool Down Mix Time = 60 minutes\*

(\*Note: Mix time reflects time required to get main batch to addition point for PEC Premix)

### [**0227**] One 11,800-kg batch

The PEC Complex in 5,678-liter / 1,500-gallon premix vessel with center turbine and side sweep agitators

main batch in M01 12,113-liter / 3,200-gallon mixing vessel with center turbine and side sweep agitators

Processing Parameters for PEC Complex:

### [0228] <u>Initial Water Charge</u> = 1840.9 L (1835 kg)

Addition Rate = 526 L/min (524 kg/min)

Total Addition Time = 3.5 minutes

Temperature =  $26^{\circ}$ C

Center Turbine Agitator = 25 RPM counter-clockwise direction

Side Sweep Agitator = 7 RPM clockwise direction

## [0229] PVM/MA Copolymer (Gantrez S-97 BF) = 23.6 kg (solid)

Addition Rate = 7.87 kg/min

Total Addition Time = 3 minutes

Temperature =  $27^{\circ}$ C

Center Turbine Agitator = 20 RPM counter-clockwise direction

Side Sweep Agitator = 7 RPM clockwise direction

Dispersion Mix Time = 30 minutes

[0230] Sodium Hydroxide, 20% = 24.2 L (29.5kg)

Addition Rate = 12.1 L/min (14.75 kg/min)

Total Addition Time = 2 minute

Temperature =  $27^{\circ}$ C

Center Turbine Agitator = 20 RPM counter-clockwise direction

Side Sweep Agitator = 7 RPM clockwise direction

Hydration Mix Time = 10 minutes

In-Process pH Check = 6.72. Add additional 118 grams (0.001 wt%) of NaOH and pH =

6.80. Add additional 118 grams (0.001 wt%) of NaOH and pH = 6.90. Total of 29.736 kg of

NaOH

Heat Premix for 18 minutes to 55°C

### [0231] Polyquaternium 28 (Conditioneze NT-20) = 1019 L (1062 kg)

Addition Rate = 29.1 L/min (30.3 kg/min)

Total Addition Time = 35 minutes

Temperature =  $54^{\circ}$ C

Center Turbine Agitator = 28 RPM counter-clockwise direction

Side Sweep Agitator = 7 RPM clockwise direction

Complex Mix Time = 30 minutes

#### [0232] Premix Cool Down

Cool to 35°C

Center Turbine Agitator = 18 RPM counter-clockwise direction

Side Sweep Agitator = 7 RPM clockwise direction

Cool Down Mix Time = 240 minutes\*

(\*Note: Mix time reflects time required to get main batch to addition point for PEC Premix)

## [0233] One 270-kg batch

The PEC Complex in 113-liter / 30-gallon premix vessel with center turbine agitator only the main batch in 227-liter / 60-gallon mixing vessel with center turbine and side sweep agitators

Processing Parameters for PEC Complex:

## [0234] Initial Water Charge = 42.04 L (41.84 kg)

Addition Rate = 28 L/min (28 kg/min)

Total Addition Time = 1.5 minutes

Initial Water Temperature = 31°C

Center Turbine Agitator = Moderate (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank-

Heat Premix for 10 minutes to 52°C

## [0235] PVM/MA Copolymer (Gantrez S-97 BF) = 0.54 kg (solid)

Addition Rate = 0.6 kg/min

Total Addition Time = 1 minute

Temperature =  $52^{\circ}$ C

Center Turbine Agitator = Moderate (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Dispersion Mix Time = 25 minutes

#### [0236] Sodium Hydroxide, 20% = 0.56 L (0.688 kg)

Addition Rate = 0.6 L/min (0.7 kg/min)

Total Addition Time = 1 minute

Temperature =  $52^{\circ}$ C

Center Turbine Agitator = Slow (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Hydration Mix Time = 15 minutes

In-Process pH Check = 6.93

## [0237] Polyquaternium 28 (Conditioneze NT-20) = 23.32 L (24.30 kg)

Addition Rate =4.66 L/min (4.86 kg/min)

Total Addition Time = 5 minutes

Temperature =  $53^{\circ}$ C

Center Turbine Agitator = Moderate/High (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Complex Mix Time = 40 minutes

### [0238] Premix Cool Down

Cool to 29°C

Center Turbine Agitator = Very Slow (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Cool Down Mix Time = 100 minutes\*

(\*Note: Mix time reflects time required to get main batch to addition point for PEC Premix)

#### [**0239**] One 270-kg batch

The PEC Complex in 113-liter / 30-gallon premix vessel with center turbine agitator only) the main batch in 227-liter / 60-gallon mixing vessel with center turbine and side sweep agitators

Processing Parameters for PEC Complex:

### [0240] Initial Water Charge = 42.03 L (41.85 kg)

Addition Rate = 28 L/min (28 kg/min)

Total Addition Time = 1.5 minutes

Initial Water Temperature = 30°C

Center Turbine Agitator = Moderate (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

## [0241] $\underline{PVM/MA Copolymer (Gantrez S-97 BF)} = 0.54 kg (solid)$

Addition Rate = 0.6 kg/min

Total Addition Time = 1 minute

Temperature =  $30^{\circ}$ C

Center Turbine Agitator = Moderate (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Dispersion Mix Time = 30 minutes

#### [0242] Sodium Hydroxide, 20% = 0.56 L (0.68 kg)

Addition Rate = 0.6 L/min (0.7 kg/min)

Total Addition Time = 1 minute

Temperature =  $31^{\circ}$ C

Center Turbine Agitator = Slow (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Hydration Mix Time = 6 minutes

In-Process pH Check = Original 6.83. Add additional 2.7 grams (0.001 wt%) of NaOH and

pH = 6.95. Total of 682.7 g of NaOH

Heat Premix for 10 minutes to 57°C

## [0243] Polyquaternium 28 (Conditioneze NT-20) = 23.32 L (24.30 kg)

Addition Rate = 3.33 L/min (3.47 kg/min)

Total Addition Time = 7 minutes

Temperature = 56°C

Center Turbine Agitator = Moderate/High (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Complex Mix Time = 30 minutes

### [0244] Premix Cool Down

Cool to 27°C

Center Turbine Agitator = Very Slow (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Cool Down Mix Time = 120 minutes\*

(\*Note: Mix time reflects time required to get main batch to addition point for PEC Premix)

### [**0245**] One 1,250-kg batch

The PEC Complex in 379-liter / 100-gallon premix vessel with center turbine and side sweep agitators

the main batch in 1,893-liter / 500-gallon mixing vessel with center turbine and side sweep agitators

Processing Parameters for PEC Complex:

[0246] Initial Water Charge = 195.2 L (194.4 kg)

Addition Rate = 97.6 L/min (97.2 kg/min)

Total Addition Time = 2 minutes

Temperature =  $29^{\circ}$ C

Center Turbine Agitator = 60 RPM counter-clockwise direction

Side Sweep Agitator = 10 RPM clockwise direction

### [0247] $\underline{PVM/MA Copolymer (Gantrez S-97 BF)} = 2.5 kg (solid)$

Addition Rate = 1.3 kg/min

Total Addition Time = 2 minutes

Temperature =  $31^{\circ}$ C

Center Turbine Agitator = 60 RPM counter-clockwise direction

Side Sweep Agitator = 10 RPM clockwise direction

Dispersion Mix Time = 26 minutes

#### [0248] Sodium Hydroxide, 20% = 2.56 L (3.125 kg)

Addition Rate = 3 L/min (4 kg/min)

Total Addition Time = 1 minute

Temperature =  $33^{\circ}$ C

Center Turbine Agitator = 20 RPM counter-clockwise direction

Side Sweep Agitator = 10 RPM clockwise direction

Hydration Mix Time = 11 minutes

In-Process pH Check = 6.90

Heat Premix for 10 minutes to 52°C

## [0249] Polyquaternium 28 (Conditioneze NT-20) = 108.0 L (112.5 kg)

Addition Rate = 9.0 L/min (9.4 kg/min)

Total Addition Time = 12 minutes

Temperature = 54°C

Center Turbine Agitator = 58 RPM counter-clockwise direction

Side Sweep Agitator = 15 RPM clockwise direction

Complex Mix Time = 31 minutes

#### [0250] Premix Cool Down

Cool to 35°C

Center Turbine Agitator = 18 RPM counter-clockwise direction

Side Sweep Agitator = 8 RPM clockwise direction

Cool Down Mix Time = 135 minutes\*

(\*Note: Mix time reflects time required to get main batch to addition point for PEC Premix)

### [**0251**] One 270-kg batch

The PEC Complex in 113-liter / 30-gallon premix vessel with center turbine agitator only the main batch in 227-liter / 60-gallon mixing vessel with center turbine and side sweep agitators

Processing Parameters for PEC Complex:

## [0252] <u>Initial Water Charge</u> = 42.01 L (41.84 kg)

Addition Rate = 28 L/min (28 kg/min)

Total Addition Time = 1.5 minutes

Initial Water Temperature = 29°C

Center Turbine Agitator = Moderate (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Heat Premix for 10 minutes to 52°C

### [0253] PVM/MA Copolymer (Gantrez S-97 BF) = 0.54 kg (solid)

Addition Rate = 0.6 kg/min

Total Addition Time = 1 minute

Temperature =  $52^{\circ}$ C

Center Turbine Agitator = Moderate (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Dispersion Mix Time = 25 minutes

### [0254] Sodium Hydroxide, 20% = 0.57 L (0.69 kg)

Addition Rate = 0.6 L/min (0.7 kg/min)

Total Addition Time = 1 minute

Temperature = 53°C

Center Turbine Agitator = Slow (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Hydration Mix Time = 5 minutes

In-Process pH Check = 6.95

## [0255] Polyquaternium 28 (Conditioneze NT-20) = 23.32 L (24.30 kg)

Addition Rate = 4.66 L/min (4.86 kg/min)

Total Addition Time = 5 minutes

Temperature = 53°C

Center Turbine Agitator = Moderate/High (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Complex Mix Time = 35 minutes

### [0256] Premix Cool Down

Cool to 30°C

Center Turbine Agitator = Very Slow (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Cool Down Mix Time = 90 minutes\*

(\*Note: Mix time reflects time required to get main batch to addition point for PEC Premix)

#### [**0257**] One 270-kg batch

The PEC Complex in 113-liter / 30-gallon premix vessel with center turbine agitator only the main batch in 227-liter / 60-gallon mixing vessel with center turbine and side sweep agitators

Processing Parameters for PEC Complex:

## [0258] <u>Initial Water Charge</u> = 42.01 L (41.84 kg)

Addition Rate = 28 L/min (28 kg/min)

Total Addition Time = 1.5 minutes

Initial Water Temperature = 29°C

Center Turbine Agitator = Moderate (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

### [0259] PVM/MA Copolymer (Gantrez S-97 BF) = 0.54 kg (solid)

Addition Rate = 0.6 kg/min

Total Addition Time = 1 minute

Temperature = 30°C

Center Turbine Agitator = Moderate (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Dispersion Mix Time = 30 minutes

## [0260] Sodium Hydroxide, 20% = 0.56 L (0.68 kg)

Addition Rate = 0.6 L/min (0.7 kg/min)

Total Addition Time = 1 minute

Temperature =  $30^{\circ}$ C

Center Turbine Agitator = Slow (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Hydration Mix Time = 20 minutes

In-Process pH Check = Original 6.81. Add additional 3.4 grams (0.00126 wt%) of NaOH

and pH = 6.92. Total of 683.4 g of NaOH

Heat Premix for 10 minutes to 54°C

### [0261] Polyquaternium 28 (Conditioneze NT-20) = 23.32 L (24.30 kg)

Addition Rate = 3.33 L/min (3.47 kg/min)

Total Addition Time = 7 minutes

Temperature = 54°C

Center Turbine Agitator = Moderate/High (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Complex Mix Time = 35 minutes

#### [0262] Premix Cool Down

Cool to 35°C

Center Turbine Agitator = Very Slow (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Cool Down Mix Time = 260 minutes\*

(\*Note: Mix time reflects time required to get main batch to addition point for PEC Premix)

### [**0263**] One 3,772-kg batch

The PEC Complex in 1,893-liter / 500-gallon premix vessel with center turbine and side sweep agitators

the main batch in 3,785-liter / 1000-gallon mixing vessel with center turbine and side sweep agitators)

Processing Parameters for PEC Complex:

### [0264] <u>Initial Water Charge</u> = 588.93 L (586.546 kg)

Addition Rate = 392.6 L/min (391 kg/min)

Total Addition Time = 1.5 minutes

Temperature =  $29^{\circ}$ C

Center Turbine Agitator = 46 RPM counter-clockwise direction

Side Sweep Agitator = 10 RPM clockwise direction

## [0265] $\underline{PVM/MA \text{ Copolymer (Gantrez S-97 BF)}} = 7.544 \text{ kg (solid)}$

Addition Rate = 3.77 kg/min

Total Addition Time = 2 minutes

Temperature =  $29^{\circ}$ C

Center Turbine Agitator = 46 RPM counter-clockwise direction

Side Sweep Agitator = 10 RPM clockwise direction

Dispersion Mix Time = 60 minutes

#### [0266] Sodium Hydroxide, 20% = 7.735 L (9.430 kg)

Addition Rate = 3.9 L/min (4.7 kg/min)

Total Addition Time = 2 minute

Temperature =  $30^{\circ}$ C

Center Turbine Agitator = 25 RPM counter-clockwise direction

Side Sweep Agitator = 10 RPM clockwise direction

Hydration Mix Time = 10 minutes

In-Process pH Check = 6.91

Heat Premix for 10 minutes to 52°C

[0267] Polyquaternium 28 (Conditioneze NT-20) = 325.80 L (339.48 kg)

Addition Rate = 16.3 L/min (17.0 kg/min)

Total Addition Time = 20 minutes

Temperature = 50°C

Center Turbine Agitator = 35 RPM counter-clockwise direction

Side Sweep Agitator = 6 RPM clockwise direction

Complex Mix Time = 40 minutes

### [0268] Premix Cool Down

Cool to 40°C

Center Turbine Agitator = 14 RPM counter-clockwise direction

Side Sweep Agitator = 6 RPM clockwise direction

Cool Down Mix Time = 55 minutes\*

(\*Note: Mix time reflects time required to get main batch to addition point for PEC Premix)

### [**0269**] One 270-kg batch

The PEC Complex in 113-liter / 30-gallon premix vessel with center turbine agitator only the main batch in 227-liter / 60-gallon mixing vessel with center turbine and side sweep agitators

Processing Parameters for PEC Complex:

[0270] Initial Water Charge = 42.01 L (41.84 kg)

Addition Rate = 28 L/min (28 kg/min)

Total Addition Time = 1.5 minutes

Initial Water Temperature = 29°C

Center Turbine Agitator = Moderate (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Heat Premix for 10 minutes to 52°C

#### [0271] PVM/MA Copolymer (Gantrez S-97 BF) = 0.54 kg (solid)

Addition Rate = 0.6 kg/min

Total Addition Time = 1 minute

Temperature = 52°C

Center Turbine Agitator = Moderate (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Dispersion Mix Time = 20 minutes

## [0272] Sodium Hydroxide, 20% = 0.57 L (0.69 kg)

Addition Rate = 0.6 L/min (0.7 kg/min)

Total Addition Time = 1 minute

Temperature =  $53^{\circ}$ C

Center Turbine Agitator = Slow (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Hydration Mix Time = 6 minutes

In-Process pH Check = 6.98

## [0273] Polyquaternium 28 (Conditioneze NT-20) = 23.32 L (24.30 kg)

Addition Rate = 4.66 L/min (4.86 kg/min)

Total Addition Time = 5 minutes

Temperature = 53°C

Center Turbine Agitator = Moderate/High (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Complex Mix Time = 35 minutes

#### [0274] Premix Cool Down

Cool to 29°C

Center Turbine Agitator = Very Slow (RPM) clockwise direction

Side Sweep Agitator = None in Pilot Premix Tank

Cool Down Mix Time = 100 minutes\*

(\*Note: Mix time reflects time required to get main batch to addition point for PEC Premix)

#### **EXAMPLE 11**

[0275] This example describes exemplary compositions of the present invention (Compositions 11A-11G). The hair mending efficacy of some of these compositions is

summarized in Tables 9A and 9B.

[0276] Composition 11A

Sprinkle in the following and mix until clear  Polyquaternium-4 (Celquat H-100)  Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate Copolymer ("Advantage S" (ISP))  Liquid Additions:  POLYQUATERNIUM-11, WATER (20% active)  VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active  Side Phase  DI Water  Slowly add the following, mix until hydrated  PVM/MA Copolymer (Gantrez S-97 BF (ISP))  0.500000  7.750000		
Sprinkle in the following and mix until clear  Polyquaternium-4 (Celquat H-100)  Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate Copolymer ("Advantage S" (ISP))  Liquid Additions:  POLYQUATERNIUM-11, WATER (20% active)  VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active  Side Phase  DI Water  Side Phase  DI Water  Side Phase  DI Water  Sodium Hydroxide (20% active)  (solution will slightly thicken)  In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	Item	wt%
Sprinkle in the following and mix until clear  Polyquaternium-4 (Celquat H-100)  Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate Copolymer ("Advantage S" (ISP))  Liquid Additions:  POLYQUATERNIUM-11, WATER (20% active)  VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active  Side Phase  DI Water  Slowly add the following, mix until hydrated PVM/MA Copolymer (Gantrez S-97 BF (ISP))  Sodium Hydroxide (20% active)  (solution will slightly thicken)  In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	Main Phase	
Polyquaternium-4 (Celquat H-100)  Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate Copolymer ("Advantage S" (ISP))  Liquid Additions:  POLYQUATERNIUM-11, WATER (20% active)  VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active  Side Phase  DI Water  Slowly add the following, mix until hydrated  PVM/MA Copolymer (Gantrez S-97 BF (ISP))  Sodium Hydroxide (20% active)  (solution will slightly thicken)  In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	DI Water	66.525000
Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate Copolymer ("Advantage S" (ISP))  Liquid Additions:  POLYQUATERNIUM-11, WATER (20% active)  VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active  Side Phase  DI Water 7.750000  Slowly add the following, mix until hydrated PVM/MA Copolymer (Gantrez S-97 BF (ISP))  Sodium Hydroxide (20% active) 0.125000  (solution will slightly thicken)  In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin 0.025000  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	Sprinkle in the following and mix until clear	
Liquid Additions:  POLYQUATERNIUM-11, WATER (20% active)  VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active  Side Phase  DI Water 7.750000  Slowly add the following, mix until hydrated PVM/MA Copolymer (Gantrez S-97 BF (ISP))  Sodium Hydroxide (20% active)  (solution will slightly thicken)  In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	Polyquaternium-4 (Celquat H-100)	0.500000
POLYQUATERNIUM-11, WATER (20% active)  VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active  Side Phase  DI Water 7.750000  Slowly add the following, mix until hydrated PVM/MA Copolymer (Gantrez S-97 BF (ISP)) 0.100000  Sodium Hydroxide (20% active) 0.125000  (solution will slightly thicken)  In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing Polyquaternium-28 (Conditioneze NT-20 (ISP)) (solution will turn opaque and become less viscous)  DMDM Hydantoin 0.025000  Slowly add side phase to main phase at ≤ 45C "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate Copolymer ("Advantage S" (ISP))	0.500000
active)  VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active  Side Phase  DI Water  Slowly add the following, mix until hydrated  PVM/MA Copolymer (Gantrez S-97 BF (ISP))  Sodium Hydroxide (20% active)  (solution will slightly thicken)  In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	Liquid Additions:	
Side Phase  DI Water  Side Phase  DI Water  Slowly add the following, mix until hydrated  PVM/MA Copolymer (Gantrez S-97 BF (ISP))  Sodium Hydroxide (20% active)  (solution will slightly thicken)  In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix		5.000000
Slowly add the following, mix until hydrated  PVM/MA Copolymer (Gantrez S-97 BF (ISP))  Sodium Hydroxide (20% active)  (solution will slightly thicken)  In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active	5.000000
Slowly add the following, mix until hydrated  PVM/MA Copolymer (Gantrez S-97 BF (ISP))  Sodium Hydroxide (20% active)  (solution will slightly thicken)  In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	Side Phase	
PVM/MA Copolymer (Gantrez S-97 BF (ISP))  Sodium Hydroxide (20% active)  (solution will slightly thicken)  In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	DI Water	7.750000
Sodium Hydroxide (20% active)  (solution will slightly thicken)  In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	Slowly add the following, mix until hydrated	Mrs. 1 page
(solution will slightly thicken)  In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.100000
In-process pH specification: 6.90-7.00  Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	Sodium Hydroxide (20% active)	0.125000
Heat to 50-55C  Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	(solution will slightly thicken)	
Slowly add the following under very vigorous mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	In-process pH specification: 6.90-7.00	
mixing  Polyquaternium-28 (Conditioneze NT-20 (ISP))  (solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix  4.500000  3.000000	Heat to 50-55C	
Conditioneze N1-20 (ISP)	•	
(solution will turn opaque and become less viscous)  DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	Polyquaternium-28 (Conditioneze NT-20 (ISP))	4.500000
DMDM Hydantoin  Slowly add side phase to main phase at ≤ 45C  "Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	(solution will turn opaque and become less	
"Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	DMDM Hydantoin	0.025000
Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)  Mix until uniform  < 45C  Silicone Premix	Slowly add side phase to main phase at $\leq 45$ C	
< 45C Silicone Premix	Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)	3.000000
Silicone Premix		
Dimethicone (60,000 cst) 2.000000		
	Dimethicone (60,000 cst)	2.000000

Phenyl Trimethicone (DC-556)	2.000000
Cyclopentasiloxane, Dimethiconol	1.000000
Glycerin	1.000000
Propylene Glycol	0.500000
Fragrance	0.300000
DMDM Hydantoin	0.175000
Citric Acid	0.000000
Sodium Hydroxide (20% active)	0.000000

# [**0277**] Composition 11B

Ĭtem	wt%
Main Phase	
DI Water	69.424700
Sprinkle in the following and mix until clear	
Polyquaternium-4 (Celquat H-100)	0.500000
Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate Copolymer ("Advantage S" (ISP))	0.500000
Liquid Additions	
POLYQUATERNIUM-11, WATER (20% active)	5.000000
VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active	5.000000
Polyquaternium-39 (8.5% active)	0.500000
Propylene Glycol	1.000000
Sorbitol (70%)	0.500000
Side Phase	
DI Water	7.750000
Slowly add the following, mix until hydrated	
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.100000
Sodium Hydroxide (20% active)	0.125000
(solution will slightly thicken)	

In-process pH specification: 6.90-7.00	
Heat Side Phase to 50-55C	
Slowly add the following under very	
vigorous mixing	
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	4.500000
(solution will turn opaque and become less viscous)	
(mix for ~10 minutes)	
Add the following at $\leq$ 45C	
DMDM Hydantoin	0.025000
Slowly add side phase to main phase at ≤ 45C	
< 45C	
Pre-mix next two and fragrance	
PEG-40 Hydrogenated Castor Oil	0.400000
PEG/PPG-18/18 Dimethicone (DC-190)	0.100000
Fragrance	0.400000
DMDM Hydantoin	0.175000
Cocos Nucifera (Coconut) Oil	0.000100
Keratin Amino Acids	0.000100
Flower/Leaf Extract	0.000100
Add the following and mix until uniform	
Polyacrylamide, C13-14 Isoparaffin, Laureth-7 ("Sepigel 305")	4.000000
Citric Acid	0.000000
Sodium Hydroxide (20% active)	0.000000

# [**0278**] Composition 11C

Item	wt%
Main Phase	
DI Water	75.725000
Sprinkle in the following and mix until clear	
Polyquaternium-4 (Celquat H-100)	0.500000

POLYQUATERNIUM-11, WATER (20% active)	2.500000
PEC Pre-mix	
DI Water	7.750000
Slowly add the following, mix until hydrated	
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.100000
Sodium Hydroxide (20% active)	0.125000
(solution will slightly thicken)	
In-process pH specification: 6.90-7.00	
Heat to 50-55C	
Slowly add the following under very vigorous mixing	
Polyquaternium-28 (Conditioneze NT-20 (ISP))	4.500000
(solution will turn opaque and become less viscous)	
mix for 10 minutes	
Add the following at $\leq 45$ C	
DMDM Hydantoin	0.025000
Slowly add side phase to main phase at $\leq 45$ C	
< 45C	
Silicone Premix	
Dimethicone (60,000 cst)	2.000000
Phenyl Trimethicone (DC-556)	2.000000
Cyclopentasiloxane, Dimethiconol	1.000000
Glycerin	1.000000
Propylene Glycol	0.500000
Fragrance	0.100000
DMDM Hydantoin	0.175000
"Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1 Trideceth-6)	2.000000
Mix until uniform	
Citric Acid	0.000000
Sodium Hydroxide (20% active)	0.000000

## [**0279**] Composition 11D

	11D-1	11D-2
Item	wt%	wt%
Main Phase		
DI Water	67.524700	55.049700
Sprinkle in the following and mix until clear		
Polyquaternium-4 (Celquat H-100)	0.500000	0.500000
Vinyl Caprolactum/VP/Dimethylaminoethyl Methacrylate		
Copolymer ("Advantage S" (ISP))	0.250000	0.250000
Disodium EDTA	0.050000	0.050000
L-Aspartic Acid	0.750000	0.750000
Add the following one at a time at 80-85C and mix until melted/homogeneous		
Stearamidopropyl Dimethylamine	2.000000	2.000000
Cetyl Alcohol	1.000000	1.000000
Glyceryl Stearate	0.200000	0.200000
Mix vigorously for 30 minutes @ 80-85C		
Reduce mixing speed; allow to cool and foam to reduce		
Liquid Additions:		
POLYQUATERNIUM-11, WATER (20% active)	1.250000	1.250000
VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active	2.500000	2.500000
Side Phase		
DI Water	7.750000	15.500000
Slowly add the following, mix until hydrated		
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.100000	0.200000
Sodium Hydroxide (20% active)	0.125000	0.250000
(solution will slightly thicken)		
In-process pH specification: 6.90-7.00		
Heat Side Phase to 50-55C		
Slowly add the following under very vigorous mixing		
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	4.500000	9.000000
(solution will turn opaque and become less viscous)		
(mix for ~10 minutes)		
Add the following at $\leq 45$ C		
DMDM Hydantoin	0.025000	0.050000
Slowly add side phase to main phase at $\leq 45$ C		
<45C		
"Abil OSW 5" - Evonik (84% Cyclopentasiloxane, 15% Dimethiconol, 1% Dimethicone)	3.000000	3.000000
"DC-8500" - Dow Corning (82% Bis(C13-15 Alkoxy) PG-Amodimethicone, 18% C14-15 Alcohols)	1.000000	1.000000
Phenyl Trimethicone (DC-556)	2.000000	2.000000

Fragrance	0.300000	0.300000
DMDM Hydantoin	0.175000	0.150000
Cocos Nucifera (Coconut) Oil	0.000100	0.000100
Keratin Amino Acids	0.000100	0.000100
Flower/Leaf Extract	0.000100	0.000100
Add the following and mix until uniform		
"Salcare SC 96" - Ciba/BASF (65% Polyquaternium-37, 25% Propylene Glycol Dicaprylate Dicaprate, 10% PPG-1		
Trideceth-6)	5.000000	5.000000
Mix until uniform		

# [**0280**] Composition 11E

Item	wt%
Main Phase	
DI Water	26.224700
Propylene Glycol	35.000000
Silicone Pre-Mix	
"Abil OSW 5" - Evonik (84% Cyclopentasiloxane, 15%	
Dimethiconol, 1% Dimethicone)	20.000000
Amodimethicone (ADM 1100 (Wacker)) (~\$10.50/kg)	2.000000
Phenyl Trimethicone (DC-556)	2.000000
Fragrance	0.100000
DMDM Hydantoin	0.175000
Cocos Nucifera (Coconut) Oil	0.000100
Keratin Amino Acids	0.000100
Flower/Leaf Extract	0.000100
Polyacrylamide, C13-14 Isoparaffin, Laureth-7 ("Sepigel 305")	2.000000
Mix moderately for ~30 minutes and until smooth	
Side Phase	
DI Water	7.750000
Slowly add the following, mix until hydrated	
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.100000
Sodium Hydroxide (20% active)	0.125000
(solution will slightly thicken)	
In-process pH specification: 6.90-7.00	
Heat Side Phase to 50-55C	
Slowly add the following under very vigorous mixing	
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	4.500000
(solution will turn opaque and become less viscous)	
(mix for ~10 minutes)	
Add the following at $\leq 45$ C	

DMDM Hydantoin	0.025000
Slowly add side phase to main phase at $\leq 45$ C	
Mix until uniform	
Citric Acid	0.000000
Sodium Hydroxide (20% active)	0.000000

## [0281] Composition 11F

Item	wt%
Main Phase	
DI Water	17.774700
Propylene Glycol	35.000000
VP/Methacrylamide/Vinyl Imidazole Copolymer ("Luviset Clear" (BASF)) - 20% active	3.750000
VP/VA Copolymer, Water	1.500000
Silicone Pre-Mix	
"Abil OSW 5" - Evonik (84% Cyclopentasiloxane, 15% Dimethiconol, 1% Dimethicone)	22.000000
Phenyl Trimethicone (DC-556)	2.000000
Fragrance	0.300000
DMDM Hydantoin	0.175000
Cocos Nucifera (Coconut) Oil	0.000100
Keratin Amino Acids	0.000100
Flower/Leaf Extract	0.000100
Polyacrylamide, C13-14 Isoparaffin, Laureth-7 ("Sepigel 305")	5.000000
Mix moderately for ~30 minutes and until smooth	
Side Phase	
DI Water	7.750000
Slowly add the following, mix until hydrated	
PVM/MA Copolymer (Gantrez S-97 BF (ISP))	0.100000
Sodium Hydroxide (20% active)	0.125000
(solution will slightly thicken)	
In-process pH specification: 6.90-7.00	
Heat Side Phase to 50-55C	
Slowly add the following under very vigorous mixing	
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	4.500000
(solution will turn opaque and become less viscous)	
(mix for ~10 minutes)	
Add the following at ≤ 45C	
DMDM Hydantoin	0.025000
Slowly add side phase to main phase at $\leq 45$ C	
Mix until uniform	

[0282] Composition 11G

	11G-1	11G-2	11G-3	11G-4
Main Phase	wt%	wt%	wt%	wt%
DI Water	76.803586	70.659300	76.803586	70.659300
Add the following under vigorous mixing, mix until hydrated (clear)				
Polyquaternium-4 (Celquat H-100)	0.543478	0.500000	0.543478	0.500000
Vinyl Caprolactum/VP/Dimethylaminoethyl				
Methacrylate Copolymer ("Advantage S"				
(ISP))	0.543478	0.500000	0.543478	0.500000
Citric Acid	0.020000	0.018400	0.020000	0.018400
Liquid Additions:				
POLYQUATERNIUM-11, WATER (21.5%				
active)	2.717391	2.500000	2.717391	2.500000
POLYQUATERNIUM-7 (8.5% active)	1.000000	0.920000	1.000000	0.920000
DC 5-7113 Silicone Quat Microemulsion -				
Dow Corning (66% Water, 25% Silicone				
Quaternium-16, 7.4% Undeceth-11, 4.5%				
Butyloctanol, 3.5% Undeceth-5)	1.000000	0.920000	1.000000	0.920000
PROPYLENE GLYCOL, USP	2.250000	2.070000	2.250000	2.070000
COCAMIDOPROPYL BETAINE (~30%				
active), WATER, SODIUM CHLORIDE	0.400000	0.368000	0.400000	0.368000
Side Phase				
DI Water	8.423913	7.750000	8.423913	7.750000
Slowly add the following, mix until hydrated				
PVM/MA Copolymer (Gantrez S-97 BF				
(ISP))	0.108696	0.100000	0.108696	0.100000
Sodium Hydroxide (20% active)	0.135870	0.125000	0.135870	0.125000
(solution will slightly thicken)				
In-process pH specification: 6.90-7.00				
Heat Side Phase to 50-55C				
Slowly add the following under very vigorous mixing				
Polyquaternium-28 (Conditioneze NT-20 (ISP), 20% active)	4.891304	4.500000	4.891304	4.500000
(solution will turn opaque and become less viscous)				
Add the following at $\leq 45$ C				
DMDM Hydantoin	0.027174	0.025	0.027174	0.025
Slowly add side phase to main phase				
Pre-Mix next and Fragrance				
POLYSORBATE 20	0.700000	0.644000	0.700000	0.644000
Fragrance	0.434783	0.400000	0.000000	0.000000
1145141100	0.000000	0.000000		+

Cocos Nucifera (Coconut) Oil	0.000109	0.000100	0.000109	0.000100
Keratin Amino Acids	0.000109	0.000100	0.000109	0.000100
Flower/Leaf Extract	0.000109	0.000100	0.000109	0.000100
Propellant Charge				
Isobutane and Propane and 152A (78.5% A46; 28.5% HFC 152A)	0.000000	8.000000	0.000000	8.000000

#### [**0283**] Table 9A

Tress#	Compos	ition 11C	Compos	ition 11A	Compos	ition 11B	Composi	tion 11E
Fiber#	3	4	5	6	7	8	9	10
1	1	1	1	1	1	1	1	1
2	1	1	1	1	1	1	1	1
3	1	0.5	1	1	1	0	0.5	1
4	1	1	1	1	1	1	1	1
5	1	1	1	0.5	1	1	1	1
6	1	1	0.5	1	1	1	1	1
7	1	0.5	1	1	1	1	1	1
8	1	1	1	1	1	1	1	1
9	1	1	1	1	0	1	1	1
10	1	1	1	1	0	1	1	1
Total	10	9	9.5	9.5	8	9	9.5	10
%								
Mended	0.	95	0.	95	0.	85	0.9	75

## [**0284**] Table 9B

Tress #	Compos	Composition 11F Composition 11G-1 Composition 11D		Composition 11G-1		ion I1D-I
Fiber #	11	12	13	14	15	16
1	0.5	1	1	1	1	1
2	1	1	1	1	1	1
3	1	1	1	1	1	1
4	1	1	1	1	1	1
5	1	1	1	1	1	0.5
6	1	0.5	1	1	1	0.5
7	1	1	1	1	1	1
8	1	1	0.5	1	1	1
9	1	1	0.5	1	1	1
10	1	1	1	1	0.5	1
Total	9.5	9.5	9	10	9.5	9
%						
Mended	0.	95	0.	95	0.9	25

[0285] All references, including publications, patent applications, and patents, cited herein are hereby incorporated by reference to the same extent as if each reference were individually and specifically indicated to be incorporated by reference and were set forth in its entirety herein.

The use of the terms "a" and "an" and "the" and similar referents in the context of [0286] describing the invention (especially in the context of the following claims) are to be construed to cover both the singular and the plural, unless otherwise indicated herein or clearly contradicted by context. The terms "comprising," "having," "including," and "containing" are to be construed as open-ended terms (i.e., meaning "including, but not limited to,") unless otherwise noted. Recitation of ranges of values herein are merely intended to serve as a shorthand method of referring individually to each separate value falling within the range, unless otherwise indicated herein, and each separate value is incorporated into the specification as if it were individually recited herein. All methods described herein can be performed in any suitable order unless otherwise indicated herein or otherwise clearly contradicted by context. The use of any and all examples, or exemplary language (e.g., "such as") provided herein, is intended merely to better illuminate the invention and does not pose a limitation on the scope of the invention unless otherwise claimed. No language in the specification should be construed as indicating any non-claimed element as essential to the practice of the invention.

[0287] Preferred embodiments of this invention are described herein, including the best mode known to the inventors for carrying out the invention. Variations of those preferred embodiments may become apparent to those of ordinary skill in the art upon reading the foregoing description. The inventors expect skilled artisans to employ such variations as appropriate, and the inventors intend for the invention to be practiced otherwise than as specifically described herein. Accordingly, this invention includes all modifications and equivalents of the subject matter recited in the claims appended hereto as permitted by applicable law. Moreover, any combination of the above-described elements in all possible variations thereof is encompassed by the invention unless otherwise indicated herein or otherwise clearly contradicted by context.

#### **CLAIMS:**

- 1. A hair care composition comprising:
  a polyelectrolyte complex comprising a cationic polymer and an anionic polymer;
  from about 1% to about 5% of a thickening agent;
  about 10% or greater of propylene glycol; and
  about 5% or greater of an emollient;
  wherein the percentages are based on the total weight of the composition.
- 2. The composition of claim 1, wherein the thickening agent comprises a quaternary ammonium nitrogen compound or polymer.
- 3. The composition of claim 2, wherein the quaternary ammonium nitrogen compound is polyquaternium-37.
- 4. The composition of claim 3, wherein the polyquaternium-37 is in a mixture with propylene glycol dicaprylate/dicaprate and polypropylene glycol-1 trideceth-6.
  - 5. The composition of claim 1, wherein the emollient is a fatty alcohol.
- 6. The composition of claim 5, wherein the fatty alcohol is cetyl alcohol, stearyl alcohol, or a combination thereof.
- 7. The composition of claim 1, wherein the polyelectrolyte complex is in an amount of from about 1% to about 8% based on the total weight of the composition.
- 8. The composition of claim 7, wherein the polyelectrolyte complex is in an amount of from about 2% to about 4% based on the total weight of the composition.
- 9. The composition of claim 1, wherein the cationic polymer comprises one or more monomeric units with one or more quaternary ammonium nitrogen moieties.
  - 10. The composition of claim 9, wherein the cationic polymer is polyquaternium-

28.

- 11. The composition of claim 1, wherein the anionic polymer comprises monomeric units having ionizable carboxylic acid moieties.
- 12. The composition of claim 11, wherein the anionic polymer is a methylvinylether/maleic acid copolymer.
- 13. The composition of claim 1, wherein the polyelectrolyte complex is in the form of a microgel.
- 14. The composition of claim 1, wherein the polyelectrolyte complex has an average particle size of 5  $\mu m$ .
- 15. The composition of claim 1, wherein the composition is in the form of a cream, a lotion, a solution, an ointment, or a gel.
- 16. The composition of claim 1, wherein the composition further comprises a preservative, a neutralizer, a fragrance, a silicone, or a combination thereof.
- 17. The composition of claim 1, wherein the cationic polymer and anionic polymer are in the weight ratio of actives is from about 1:8 to about 1:10.
- 18. A method of conditioning a hair fiber, the method comprising applying to the hair fiber a composition of claim 1 and rinsing the hair fiber with water.
- 19. The method of claim 18, wherein the hair fiber is dried using a blow dryer after rinsing.
- 20. A method of repairing a split-end of a hair fiber, the method comprising applying to the split-end a composition of claim 1 and rinsing the split-end with water.
  - 21. The method of claim 20, wherein the split-end is dried using a blow dryer after

rinsing.

- 22. A method for improving the freeze-thaw stability of a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, the method comprising adding to the hair care composition from about 1% to about 5% of a thickening agent, wherein the percentage is based on the total weight of the composition.
- 23. The method of claim 22, wherein the thickening agent comprises a quaternary ammonium nitrogen compound or polymer.
- 24. The method of claim 23, wherein the quaternary ammonium nitrogen compound is polyquaternium-37.
- 25. The method of claim 24, wherein the polyquaternium-37 is in a mixture with propylene glycol dicaprylate/dicaprate and polypropylene glycol-1 trideceth-6.
- 26. A method for improving the freeze-thaw stability of a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, the method comprising adding to the hair care composition from about 10% or greater of propylene glycol, wherein the percentage is based on the total weight of the composition.
  - 27. A hair care composition comprising:
    a polyelectrolyte complex comprising a cationic polymer and an anionic polymer;
    from about 3% to about 5% of a thickening agent; and
    about 1% or less of an emollient;
    wherein the percentages are based on the total weight of the composition.
- 28. The composition of claim 27, wherein the thickening agent comprises a quaternary ammonium nitrogen compound or polymer.
- 29. The composition of claim 28, wherein the quaternary ammonium nitrogen compound is polyquaternium-37.

- 30. The composition of claim 29, wherein the polyquaternium-37 is in a mixture with propylene glycol dicaprylate/dicaprate and polypropylene glycol-1 trideceth-6.
  - 31. The composition of claim 27, wherein the emollient is a fatty alcohol.
- 32. The composition of claim 31, wherein the fatty alcohol is cetyl alcohol, stearyl alcohol, or a combination thereof.
- 33. The composition of claim 27, wherein the polyelectrolyte complex is in an amount of from about 1% to about 8% based on the total weight of the composition.
- 34. The composition of claim 33, wherein the polyelectrolyte complex is in an amount of from about 2% to about 4% based on the total weight of the composition.
- 35. The composition of claim 27, wherein the cationic polymer comprises one or more monomeric units with one or more quaternary ammonium nitrogen moieties.
- 36. The composition of claim 35, wherein the cationic polymer is polyquaternium-28.
- 37. The composition of claim 27, wherein the anionic polymer comprises monomeric units having ionizable carboxylic acid moieties.
- 38. The composition of claim 37, wherein the anionic polymer is a methylvinylether/maleic acid copolymer.
- 39. The composition of claim 27, wherein the polyelectrolyte complex is in the form of a microgel.
- 40. The composition of claim 27, wherein the polyelectrolyte complex has an average particle size of 5  $\mu m$ .
  - 41. The composition of claim 27, wherein the composition is in the form of a

cream, a lotion, a solution, an ointment, or a gel.

- 42. The composition of claim 27, wherein the composition further comprises a preservative, a neutralizer, a fragrance, a silicone, or a combination thereof.
- 43. The composition of claim 27, wherein the cationic polymer and anionic polymer are in the weight ratio of actives is from about 1:8 to about 1:10.
- 44. A method of conditioning a hair fiber, the method comprising applying to the hair fiber a composition of claim 27 and rinsing the hair fiber with water.
- 45. The method of claim 44, wherein the hair fiber is dried using a blow dryer after rinsing.
- 46. A method of repairing a split-end of a hair fiber, the method comprising applying to the split-end a composition of claim 27 and rinsing the split-end with water.
- 47. The method of claim 46, wherein the split-end is dried using a blow dryer after rinsing.
- 48. A hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, wherein the viscosity of the composition is greater than 3000 cps.
- 49. The composition of claim 48, wherein the polyelectrolyte complex is in an amount of from about 1% to about 8% based on the total weight of the composition.
- 50. The composition of claim 49, wherein the polyelectrolyte complex is in an amount of from about 2% to about 4% based on the total weight of the composition.
- 51. The composition of claim 48, wherein the cationic polymer comprises one or more monomeric units with one or more quaternary ammonium nitrogen moieties.

- 52. The composition of claim 51, wherein the cationic polymer is polyquaternium-28.
- 53. The composition of claim 48, wherein the anionic polymer comprises monomeric units having ionizable carboxylic acid moieties.
- 54. The composition of claim 53, wherein the anionic polymer is a methylvinylether/maleic acid copolymer.
- 55. The composition of claim 48, wherein the polyelectrolyte complex is in the form of a microgel.
- 56. The composition of claim 48, wherein the polyelectrolyte complex has an average particle size of 5  $\mu m$ .
- 57. The composition of claim 48, wherein the composition is in the form of a cream, a lotion, a solution, an ointment, or a gel.
- 58. The composition of claim 48, wherein the cationic polymer and anionic polymer are in the weight ratio of actives is from about 1:8 to about 1:10.
- 59. The composition of claim 48, wherein the composition further comprises a preservative, a neutralizer, a fragrance, a silicone, or a combination thereof.
  - 60. The composition of claim 48, wherein the composition further comprises from about 1% to about 5% of a thickening agent; about 10% or greater of propylene glycol; and about 5% or greater of an emollient; wherein the percentages are based on the total weight of the composition.
  - 61. The composition of claim 48, wherein the composition further comprises from about 3% to about 5% of a thickening agent; and about 1% or less of an emollient;

wherein the percentages are based on the total weight of the composition.

- 62. A method of conditioning a hair fiber, the method comprising applying to the hair fiber a composition of claim 48 and rinsing the hair fiber with water.
- 63. The method of claim 62, wherein the hair fiber is dried using a blow dryer after rinsing.
- 64. A method of repairing a split-end of a hair fiber, the method comprising applying to the split-end a composition of claim 48 and rinsing the split-end with water.
- 65. The method of claim 64, wherein the split-end is dried using a blow dryer after rinsing.
- 66. A hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer in the form of an emulsion with improved freeze-thaw stability relative to comparative compositions containing such polyelectrolyte complexes.
- 67. The composition of claim 66, wherein the polyelectrolyte complex is in an amount of from about 1% to about 8% based on the total weight of the composition.
- 68. The composition of claim 67, wherein the polyelectrolyte complex is in an amount of from about 2% to about 4% based on the total weight of the composition.
- 69. The composition of claim 66, wherein the cationic polymer comprises one or more monomeric units with one or more quaternary ammonium nitrogen moieties.
- 70. The composition of claim 69, wherein the cationic polymer is polyquaternium-28.
- 71. The composition of claim 66, wherein the anionic polymer comprises monomeric units having ionizable carboxylic acid moieties.

- 72. The composition of claim 71, wherein the anionic polymer is a methylvinylether/maleic acid copolymer.
- 73. The composition of claim 66, wherein the polyelectrolyte complex is in the form of a microgel.
- 74. The composition of claim 66, wherein the polyelectrolyte complex has an average particle size of 5  $\mu$ m.
- 75. The composition of claim 66, wherein the composition is in the form of a cream, a lotion, a solution, an ointment, or a gel.
- 76. The composition of claim 66, wherein the cationic polymer and anionic polymer are in the weight ratio of actives is from about 1:8 to about 1:10.
- 77. The composition of claim 66, wherein the composition further comprises a preservative, a neutralizer, a fragrance, a silicone, or a combination thereof.
  - 78. The composition of claim 66, wherein the composition further comprises from about 1% to about 5% of a thickening agent; about 10% or greater of propylene glycol; and about 5% or greater of an emollient; wherein the percentages are based on the total weight of the composition.
  - 79. The composition of claim 66, wherein the composition further comprises from about 3% to about 5% of a thickening agent; and about 1% or less of an emollient; wherein the percentages are based on the total weight of the composition.
- 80. A method of conditioning a hair fiber, the method comprising applying to the hair fiber a composition of claim 66 and rinsing the hair fiber with water.
  - 81. The method of claim 80, wherein the hair fiber is dried using a blow dryer

after rinsing.

- 82. A method of repairing a split-end of a hair fiber, the method comprising applying to the split-end a composition of claim 66 and rinsing the split-end with water.
- 83. The method of claim 82, wherein the split-end is dried using a blow dryer after rinsing.
- 84. A method of producing a polyelectrolyte complex, the method comprising: hydrating a methylvinylether/maleic acid copolymer having repeating units of the formula:

in a container;

reacting the methylvinylether/maleic acid copolymer with a base in the container in an amount sufficient to at least partially neutralize the acidic groups thereof, to form an intermediate mixture;

heating the intermediate mixture in the container;

adding to the intermediate mixture in the container a cationic copolymer of a vinylpyrrolidone of the formula:

and a methacrylamidopropyl trimethylammonium chloride of the formula:

; and

mixing the polymers in the container at high shear force and at a temperature of at least about 50-60 °C to produce the polyelectrolyte complex.

- 85. The method of claim 84, wherein the base is a 20% active solution of sodium hydroxide.
- 86. The method of claim 85, wherein the sodium hydroxide is added in an amount sufficient to achieve a pH of from about 6.5 to about 7.5.
- 87. The method of claim 86, wherein the sodium hydroxide is added in an amount sufficient to achieve a pH of from about 6.9 to about 7.0.
- 88. The method of claim 84, wherein the intermediate mixture is heated to a temperature of from about 50 °C to about 60 °C.
- 89. The method of claim 84, wherein the cationic copolymer is added to the intermediate mixture at from about 0.3 kg/min per minute to about 1.0 kg/min per minute.
- 90. The method of claim 84, wherein the weight ratio of actives of the methylvinylether/maleic acid copolymer to the cationic copolymer of a vinylpyrrolidone and a methacrylamidopropyl trimethylammonium chloride is from about 1:8 to about 1:10.
- 91. The method of claim 84, wherein the resulting polyelectrolyte complex is at a concentration of greater than 4 wt. %.
- 92. The method of claim 84, wherein the intermediate mixture is mixed for at least about 10 minutes prior to addition of the cationic polymer.
- 93. The method of claim 84, wherein the particle size of the polyelectrolyte complex averages  $5 \pm 2$  micrometers.

- 94. The method of claim 84, further comprising the step of mixing the polyelectrolyte complex with one or more components selected from aqueous carriers, surfactants, fragrances, conditioning agents, emollients, emulsifiers, thickening agents, preservatives, gelling agents, silicones, hair fixatives, humectants and moisturizers to form a hair care composition.
- 95. The method of claim 94, wherein the mixing is sufficient to allow adequate incorporation of the one or more components.
- 96. A method of producing a polyelectrolyte complex, the method comprising: hydrating a methylvinylether/maleic acid copolymer having repeating units of the formula:

in a container;

reacting the methylvinylether/maleic acid copolymer with a 20% sodium hydroxide solution in the container, wherein the sodium hydroxide is added in an amount sufficient to achieve a pH of from about 6.9 to about 7.0, to form an intermediate mixture;

heating the intermediate mixture in the container to a temperature of from about 50  $^{\circ}$ C to about 60  $^{\circ}$ C;

adding to the intermediate mixture in the container a cationic copolymer of a vinylpyrrolidone of the formula:

and a methacrylamidopropyl trimethylammonium chloride of the formula:

; and

mixing the polymers in the container for at least about 10 minutes at high shear force and at a temperature of at least about 50-60 °C to produce the polyelectrolyte complex.

- 97. The method of claim 96, wherein the cationic copolymer is added to the intermediate mixture at from about 0.3 kg/min per minute to about 1.0 kg/min per minute.
- 98. The method of claim 96, wherein the weight ratio of actives of the methylvinylether/maleic acid copolymer to the cationic copolymer of a vinylpyrrolidone and a methacrylamidopropyl trimethylammonium chloride is from about 1:8 to about 1:10.
- 99. The method of claim 96, wherein the resulting polyelectrolyte complex is at a concentration of greater than 4 wt. %.
- 100. The method of claim 96, wherein the intermediate mixture is mixed for at least about 10 minutes prior to addition of the cationic polymer.
- 101. The method of claim 96, wherein the particle size of the polyelectrolyte complex averages  $5 \pm 2$  micrometers.
- 102. The method of claim 96, further comprising the step of mixing the polyelectrolyte complex with one or more components selected from aqueous carriers, surfactants, fragrances, conditioning agents, emollients, emulsifiers, thickening agents, preservatives, gelling agents, silicones, hair fixatives, humectants and moisturizers to form a hair care composition.
- 103. The method of claim 102, wherein the mixing is sufficient to allow adequate incorporation of the one or more components.
  - 104. A method of producing a hair care composition comprising: producing a polyelectrolyte complex, comprising the steps of:

hydrating a methylvinylether/maleic acid copolymer having repeating units of the formula:

in a container;

reacting the methylvinylether/maleic acid copolymer with a base in the container in an amount sufficient to at least partially neutralize the acidic groups thereof, to form an intermediate mixture;

heating the intermediate mixture in the container;

adding to the intermediate mixture in the container a cationic copolymer of a vinylpyrrolidone of the formula:

and a methacrylamidopropyl trimethylammonium chloride of the formula:

: and

mixing the polymers in the container at high shear force and at a temperature of at least about 50-60 °C to produce the polyelectrolyte complex;

adding to the polyelectrolyte complex in the container one or more additional components selected from aqueous carriers, surfactants, fragrances, conditioning agents, emollients, emulsifiers, thickening agents, preservatives, gelling agents, silicones, hair fixatives, humectants and moisturizers; and

mixing the resulting composition in the container.

- 105. The method of claim 104, wherein the base is a 20% active solution of sodium hydroxide.
- 106. The method of claim 105, wherein the sodium hydroxide is added in an amount sufficient to achieve a pH of from about 6.5 to about 7.5.
- 107. The method of claim 106, wherein the sodium hydroxide is added in an amount sufficient to achieve a pH of from about 6.9 to about 7.0.
- 108. The method of claim 104, wherein the intermediate mixture is heated to a temperature of from about 50  $^{\circ}$ C to about 60  $^{\circ}$ C.
- 109. The method of claim 104, wherein the cationic copolymer is added to the intermediate mixture at from about 0.3 kg/min per minute to about 1.0 kg/min per minute.
- 110. The method of claim 104, wherein the weight ratio of actives of the methylvinylether/maleic acid copolymer to the cationic copolymer of a vinylpyrrolidone and a methacrylamidopropyl trimethylammonium chloride is from about 1:8 to about 1:10.
- 111. The method of claim 104, wherein the resulting polyelectrolyte complex is at a concentration of greater than 4 wt. %.
- 112. The method of claim 104, wherein the intermediate mixture is mixed for at least about 10 minutes prior to addition of the cationic polymer.
- 113. The method of claim 104, wherein the particle size of the polyelectrolyte complex averages  $5 \pm 2$  micrometers.
- 114. The method of claim 104, wherein the hair care composition is in the form of a shampoo, conditioner, leave-in treatment, crème, gel, mousse, or spray.
- 115. The method of claim 104, wherein the one or more additional components is one or more fatty alcohols.
- 116. The method of claim 115, wherein the one or more fatty alcohols are selected from cetyl alcohol and stearyl alcohol.

- 117. The method of claim 104, wherein the one or more additional components is polyquaternium-37.
- 118. The method of claim 104, wherein the one or more additional components is one or more cleansing surfactants.
- 119. The method of claim 118, wherein the one or more cleansing surfactants are selected from sodium laureth sulfate, disodium laureth sulfosuccinate, and cocamidopropyl betaine.
- 120. The method of claim 104, wherein the one or more additional components is one or more silicones.
- 121. The method of claim 120, wherein the one or more silicones are selected from dimethicone, phenyl trimethicone, and dimethiconol.
- 122. The method of claim 104, wherein the one or more additional components is propylene glycol.
  - 123. A method of producing a hair care composition comprising:

producing a polyelectrolyte complex, comprising the steps of:

hydrating a methylvinylether/maleic acid copolymer having repeating units of the formula:

in a first container;

reacting the methylvinylether/maleic acid copolymer with a base in the first container in an amount sufficient to at least partially neutralize the acidic groups thereof, to form an intermediate mixture;

heating the intermediate mixture in the first container;

adding to the intermediate mixture in the first container a cationic copolymer of a vinylpyrrolidone of the formula:

and a methacrylamidopropyl trimethylammonium chloride of the formula:

; and

mixing the polymers in the first container at high shear force and at a temperature of at least about 50-60 °C to produce the polyelectrolyte complex;

mixing in a second container one or more components selected from aqueous carriers, surfactants, fragrances, conditioning agents, emollients, emulsifiers, thickening agents, preservatives, gelling agents, silicones, hair fixatives, humectants and moisturizers; and

mixing together in the first or second container the polyelectrolyte complex of the first container and the mixed components of the second container.

- 124. The method of claim 123, wherein the base is a 20% active solution of sodium hydroxide.
- 125. The method of claim 124, wherein the sodium hydroxide is added in an amount sufficient to achieve a pH of from about 6.5 to about 7.5.
- 126. The method of claim 125, wherein the sodium hydroxide is added in an amount sufficient to achieve a pH of from about 6.9 to about 7.0.
- 127. The method of claim 123, wherein the intermediate mixture is heated to a temperature of from about 50 °C to about 60 °C.
  - 128. The method of claim 123, wherein the cationic copolymer is added to the

intermediate mixture at from about 0.3 kg/min per minute to about 1.0 kg/min per minute.

- 129. The method of claim 123, wherein the weight ratio of actives of the methylvinylether/maleic acid copolymer to the cationic copolymer of a vinylpyrrolidone and a methacrylamidopropyl trimethylammonium chloride is from about 1:8 to about 1:10.
- 130. The method of claim 123, wherein the resulting polyelectrolyte complex is at a concentration of greater than 4 wt. %.
- 131. The method of claim 123, wherein the intermediate mixture is mixed for at least about 10 minutes prior to addition of the cationic polymer.
- 132. The method of claim 123, wherein the particle size of the polyelectrolyte complex averages  $5 \pm 2$  micrometers.
- 133. The method of claim 123, wherein the hair care composition is in the form of a shampoo, conditioner, leave-in treatment, crème, gel, mousse, or spray.
- 134. The method of claim 123, wherein the one or more additional components is one or more fatty alcohols.
- 135. The method of claim 134, wherein the one or more fatty alcohols are selected from cetyl alcohol and stearyl alcohol.
- 136. The method of claim 123, wherein the one or more additional components is polyquaternium-37.
- 137. The method of claim 123, wherein the one or more additional components is one or more cleansing surfactants.
- 138. The method of claim 137, wherein the one or more cleansing surfactants are selected from sodium laureth sulfate, disodium laureth sulfosuccinate, and cocamidopropyl betaine.
- 139. The method of claim 123, wherein the one or more additional components is one or more silicones.
  - 140. The method of claim 139, wherein the one or more silicones are selected from

dimethicone, phenyl trimethicone, and dimethiconol.

- 141. The method of claim 123, wherein the one or more additional components is propylene glycol.
- 142. A method of quantitatively assessing the degree of split-end hair fiber repair, the method comprising:

obtaining a tress of hair, wherein the hair is twice-bleached hair;

combing and blow drying the tress of hair to produce at least one spilt-end in at least one hair fiber;

removing at least one hair fiber having a split-end from the tress;

optionally marking the at least one hair fiber;

capturing a first image of the split-end of the at least one hair fiber and storing a representation of the image;

associating the hair fiber with the tress from which the hair fiber was removed;

treating the tress and the hair fiber with a composition;

removing the hair fiber from the treated tress;

capturing a second image of the end of the hair fiber removed from the treated tress; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for

complete repair with respect to the hair fiber to assess the degree of split-end repair of the hair fiber; and

if more than one hair fiber is assessed, optionally determining an average value with respect to the assessed hair fibers.

- 143. The method of claim 142, wherein the hair is human hair.
- 144. The method of claim 143, wherein the then human hair is European hair.
- 145. The method of claim 142, wherein the composition comprises a polyelectrolyte complex of a cationic polymer and an anionic polymer.
- 146. The method of claim 145, wherein the polyelectrolyte complex is in an amount of from about 1% to about 8% based on the total weight of the composition.

- 147. The method of claim 146, wherein the polyelectrolyte complex is in an amount of from about 2% to about 4% based on the total weight of the composition.
- 148. The method of claim 145, wherein the cationic polymer comprises one or more monomeric units with one or more quaternary ammonium nitrogen moieties.
  - 149. The method of claim 148, wherein the cationic polymer is polyquaternium-28.
- 150. The method of claim 145, wherein the anionic polymer comprises monomeric units having ionizable carboxylic acid moieties.
- 151. The method of claim 150, wherein the anionic polymer is a methylvinylether/maleic acid copolymer.
- 152. The method of claim 145, wherein the polyelectrolyte complex is in the form of a microgel.
- 153. The method of claim 145, wherein the polyelectrolyte complex has an average particle size of 5  $\mu m$ .
- 154. The method of claim 142, wherein the composition is in the form of a cream, a lotion, a solution, an ointment, or a gel.
- 155. The method of claim 145, wherein the cationic polymer and anionic polymer are in the weight ratio of actives is from about 1:8 to about 1:10.
- 156. The method of claim 145, wherein the composition further comprises a preservative, a neutralizer, a fragrance, a silicone, or a combination thereof.
  - 157. The method of claim 145, wherein the composition further comprises from about 1% to about 5% of a thickening agent; about 10% or greater of propylene glycol; and about 5% or greater of an emollient;

wherein the percentages are based on the total weight of the composition.

- 158. The method of claim 145, wherein the composition further comprises from about 3% to about 5% of a thickening agent; and about 1% or less of an emollient; wherein the percentages are based on the total weight of the composition.
- 159. The method of claim 142, wherein the degree of split-end hair fiber repair is communicated to consumers.
- 160. A method of quantitatively assessing the degree of split-end hair fiber repair, the method comprising:

obtaining a tress of hair;

producing at least one hair fiber of the tress to have a spilt-end;

removing at least one hair fiber having a split-end from the tress;

optionally marking the at least one hair fiber;

capturing a first image of the split-end of the at least one hair fiber and storing a representation of the image;

associating the hair fiber with the tress from which the hair fiber was removed; treating the tress and the hair fiber with a composition, wherein the composition comprises a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, from about 1% to about 5% of a thickening agent, about 10% or greater of propylene glycol, and about 5% or greater of an emollient, wherein the percentages are based on the total weight of the composition;

removing the hair fiber from the treated tress;

capturing a second image of the end of the hair fiber removed from the treated tress; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair with respect to the hair fiber to assess the degree of split-end repair of the hair fiber; and

if more than one hair fiber is assessed, optionally determining an average value with respect to the assessed hair fibers.

161. A method of quantitatively assessing the degree of split-end hair fiber repair,

the method comprising:

obtaining a tress of hair;

producing at least one hair fiber of the tress to have a spilt-end;

removing at least one hair fiber having a split-end from the tress;

optionally marking the at least one hair fiber;

capturing a first image of the split-end of the at least one hair fiber and storing a representation of the image;

associating the hair fiber with the tress from which the hair fiber was removed; treating the tress and the hair fiber with a composition, wherein the composition comprises a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer, from about 3% to about 5% of a thickening agent, and about 1% or less of an emollient, wherein the percentages are based on the total weight of the composition;

removing the hair fiber from the treated tress;

capturing a second image of the end of the hair fiber removed from the treated tress; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair with respect to the hair fiber to assess the degree of split-end repair of the hair fiber; and

if more than one hair fiber is assessed, optionally determining an average value with respect to the assessed hair fibers.

162. A method of quantitatively assessing the degree of split-end hair fiber repair, the method comprising:

obtaining a tress of hair;

producing at least one hair fiber of the tress to have a spilt-end;

removing at least one hair fiber having a split-end from the tress;

optionally marking the at least one hair fiber;

capturing a first image of the split-end of the at least one hair fiber and storing a representation of the image;

associating the hair fiber with the tress from which the hair fiber was removed; treating the tress and the hair fiber with a composition, wherein the composition

polymer and an anionic polymer, wherein the viscosity of the composition is greater than

comprises a hair care composition comprising a polyelectrolyte complex of a cationic

3000 cps;

removing the hair fiber from the treated tress;

capturing a second image of the end of the hair fiber removed from the treated tress; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair with respect to the hair fiber to assess the degree of split-end repair of the hair fiber; and

if more than one hair fiber is assessed, optionally determining an average value with respect to the assessed hair fibers.

163. A method of quantitatively assessing the degree of split-end hair fiber repair, the method comprising:

obtaining a tress of hair;

producing at least one hair fiber of the tress to have a spilt-end;

removing at least one hair fiber having a split-end from the tress;

optionally marking the at least one hair fiber;

capturing a first image of the split-end of the at least one hair fiber and storing a representation of the image;

associating the hair fiber with the tress from which the hair fiber was removed; treating the tress and the hair fiber with a composition, wherein the composition comprises a hair care composition comprising a polyelectrolyte complex of a cationic polymer and an anionic polymer in the form of an emulsion with improved freeze-thaw stability relative to comparative compositions containing such polyelectrolyte complexes;

removing the hair fiber from the treated tress;

capturing a second image of the end of the hair fiber removed from the treated tress; assigning a value of 0 for no repair of the split-end, ½ for partial repair, or 1 for complete repair with respect to the hair fiber to assess the degree of split-end repair of the hair fiber; and

if more than one hair fiber is assessed, optionally determining an average value with respect to the assessed hair fibers.

164. A method of graphically reconstructing a repair of a split-end of a hair fiber, the method comprising:

securing a hair fiber to a surface, wherein the hair fiber secured to the surface has a

split-end or is damaged while on the surface to produce a split-end;

associating the surface with a magnifying device, wherein the split-end is positioned within the field of view of the magnifying device;

associating an image capture device with the magnifying device so as to capture an enlarged image of the field of view of the magnifying device;

treating the spilt-end with a composition, wherein the composition repairs the splitend and wherein the split-end fuses together; and

via the image capture device associated with the magnifying device, capturing an image of the repair of the split-end during treatment with the composition, and storing a representation of the captured image of the repair for subsequent retrieval.

- 165. The method of claim 164, wherein the image capture device is a digital recorder.
- 166. The method of claim 164, wherein the magnifying device is a light microscope.
  - 167. The method of claim 164, wherein the hair is human hair.
  - 168. The method of claim 167, wherein the then human hair is European hair.
- 169. The method of claim 164, wherein the composition comprises a polyelectrolyte complex of a cationic polymer and an anionic polymer.
- 170. The method of claim 169, wherein the polyelectrolyte complex is in an amount of from about 1% to about 8% based on the total weight of the composition.
- 171. The method of claim 170, wherein the polyelectrolyte complex is in an amount of from about 2% to about 4% based on the total weight of the composition.
- 172. The method of claim 169, wherein the cationic polymer comprises one or more monomeric units with one or more quaternary ammonium nitrogen moieties.

- 173. The method of claim 172, wherein the cationic polymer is polyquaternium-28.
- 174. The method of claim 169, wherein the anionic polymer comprises monomeric units having ionizable carboxylic acid moieties.
- 175. The method of claim 174, wherein the anionic polymer is a methylvinylether/maleic acid copolymer.
- 176. The method of claim 169, wherein the polyelectrolyte complex is in the form of a microgel.
- 177. The method of claim 169, wherein the polyelectrolyte complex has an average particle size of 5  $\mu m$ .
- 178. The method of claim 164, wherein the composition is in the form of a cream, a lotion, a solution, an ointment, or a gel.
- 179. The method of claim 169, wherein the cationic polymer and anionic polymer are in the weight ratio of actives is from about 1:8 to about 1:10.
- 180. The method of claim 169, wherein the composition further comprises a preservative, a neutralizer, a fragrance, a silicone, or a combination thereof.
  - 181. The method of claim 169, wherein the composition further comprises from about 1% to about 5% of a thickening agent; about 10% or greater of propylene glycol; and about 5% or greater of an emollient; wherein the percentages are based on the total weight of the composition.
  - 182. The method of claim 169, wherein the composition further comprises from about 3% to about 5% of a thickening agent; and about 1% or less of an emollient; wherein the percentages are based on the total weight of the composition.

- 183. The method of claim 164, wherein the degree of split-end hair fiber repair is communicated to consumers.
  - 184. The method of claim 164, wherein the image capture device is a video camera.
- 185. The method of claim 184, wherein the method comprises recording the hair reparation in real-time.

Figure 1

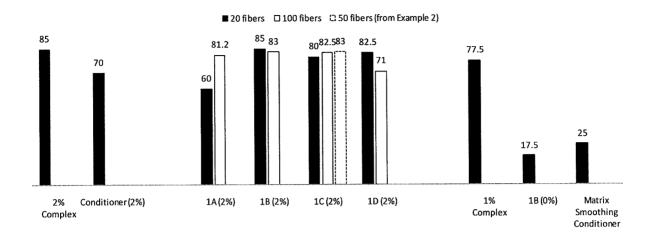


Figure 2

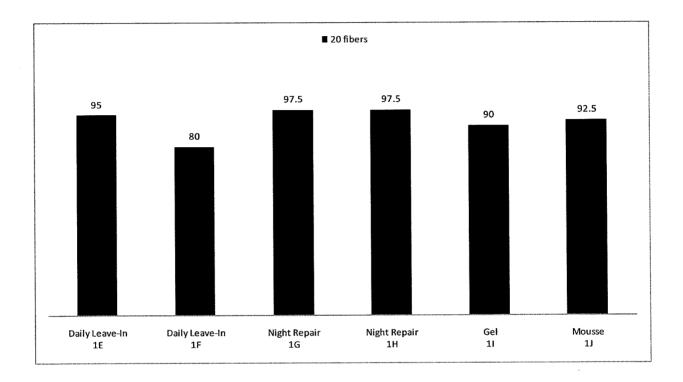


Figure 3

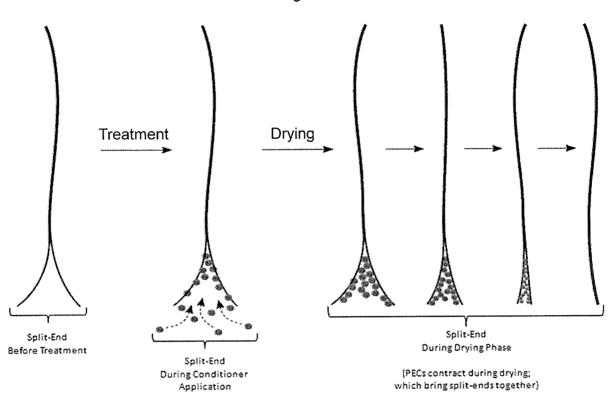
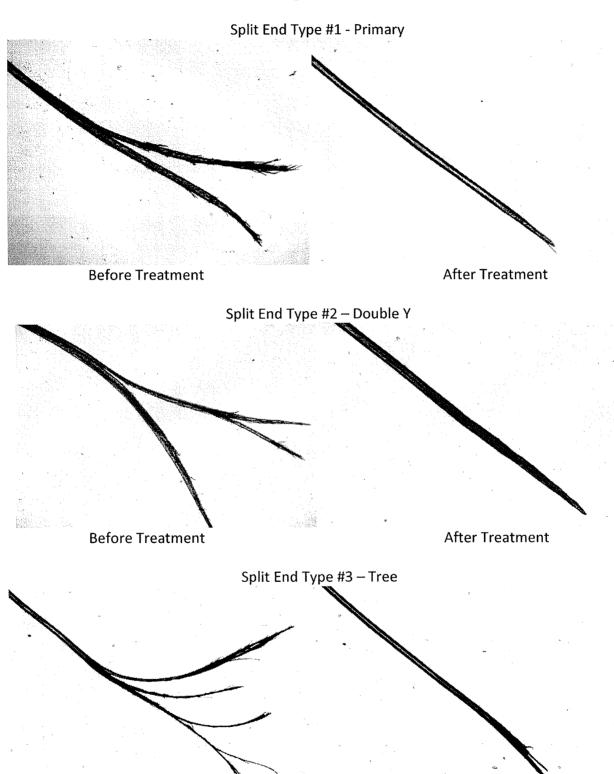


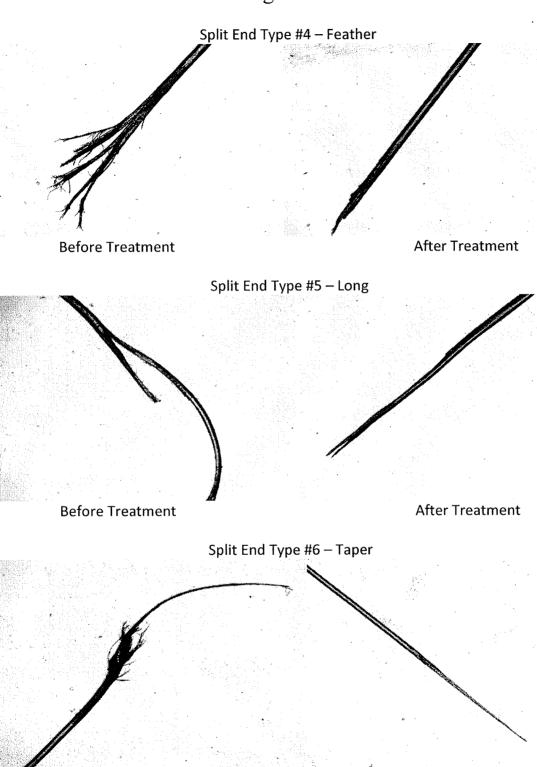
Figure 4A



After Treatment

**Before Treatment** 

Figure 4B



After Treatment

**Before Treatment** 

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Figure 4C

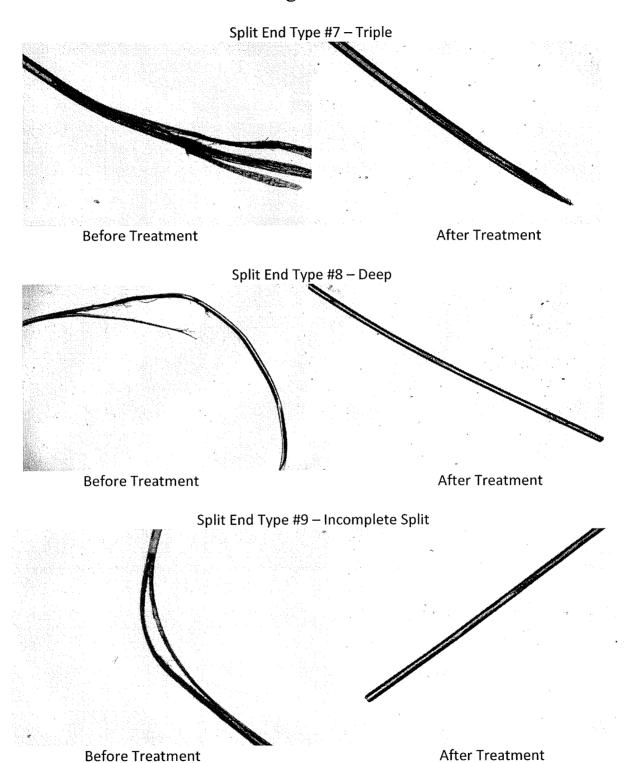
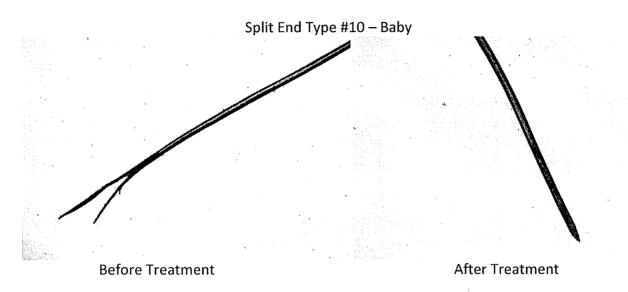


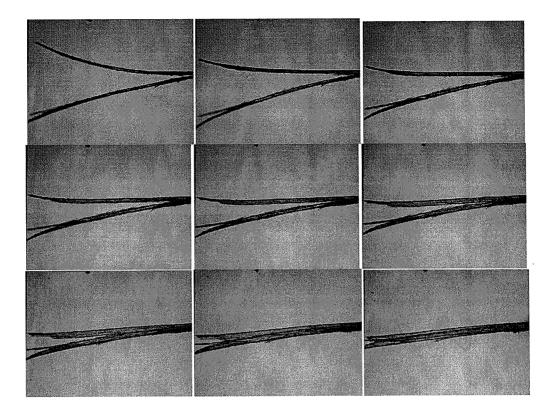
Figure 4D



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Figure 5



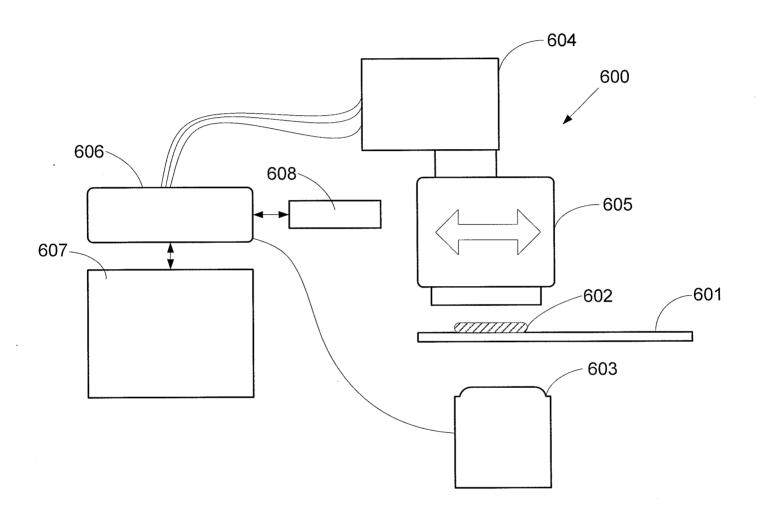


FIG. 6

#### INTERNATIONAL SEARCH REPORT

International application No. PCT/US 10/53360

IPC(8) - USPC -	SSIFICATION OF SUBJECT MATTER A61K 8/72; A61K 9/14 (2010.01) 424/489; 424/70.11 o International Patent Classification (IPC) or to both n	ational classification and IPC					
B. FIELDS SEARCHED							
	ocumentation searched (classification system followed by 489; 424/70.11	classification symbols)					
	ion searched other than minimum documentation to the ex 70.11, 424/70.13, 424/70.15, 424/70.16, 424/489; 510/		fields searched				
*** Database	ata base consulted during the international search (name of the search (name of the search (PGPB, USPT, USOC, EPAB, JPAB); Google erms Used: Alberto-Culver, Wright, Szerszen, Cohen, Furm, 28, 37, propylene glycol, thickening agent, thickene	o Petroski, Eagan, Felski, polyelectrolyte, com	nplex, hair, split end,				
C. DOCU	MENTS CONSIDERED TO BE RELEVANT						
Category*	Citation of document, with indication, where a	opropriate, of the relevant passages	Relevant to claim No.				
X  Y	US 2006/0188468 A1 (Nguyen-Kim et al.) 24 August 2 [0020]-[0022], [0028], [0036], [0123], [0134], [0164], [0 [0220], [0232], [0235]-[0236], [0272]-[0273], [0238], [0285]-[0286], [0289], [0293], [0317]-[0322]	167], [0181]-[0182], [0192], [0200],	1-9, 11-12, 15-19, 22-35, 37-38, 41-45, 48-51, 53-54, 57-63, 66-69, 71-72, 75-81				
			10, 13-14, 20-21, 36, 39- 40, 46-47, 52, 55-56, 64- 65, 70, 73-74, 82-141, 155, 157-158, 160-163, 179, 181-182				
X  Y	US 2006/0251603 A1 (Rigoletto et al.) 09 November 2 [0003], [0022]-[0024], [0026]-[0030], [0033]-[0036], [00 [0103]		142-154, 156, 159, 164- 178, 180, 183-184 				
Α	US 2005/0089494 A1 (Rigoletto) 28 April 2005 (28.04.	2005), entire document	1-184				
A	Rigoletto et al., "Semi-permanent split end mending wi Journal of Cosmetic Science, Vol. 58, Is. 4, Pg. 451-47		1-184				
Furthe	er documents are listed in the continuation of Box C.						
"A" docume to be of	* Special categories of cited documents:  "A" document defining the general state of the art which is not considered to be of particular relevance  "I" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention						
"E" earlier application or patent but published on or after the international filing date "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone							
cited to establish the publication date of another citation or other special reason (as specified)  "Y"  document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is							
"O" document referring to an oral disclosure, use, exhibition or other means the such documents, such combination being obvious to a person skilled in the art							
the priority date claimed							
	28 February 2011 (28.02.2011) 07 MAR 2011						
	nailing address of the ISA/US	Authorized officer:					
	Aail Stop PCT, Attn: ISA/US, Commissioner for Patents  1.O. Box 1450, Alexandria, Virginia 22313-1450						
	0. 571-273-3201	PCT Helpdesk: 571-272-4300 PCT OSP: 571-272-7774					

Form PCT/ISA/210 (second sheet) (July 2009)

#### INTERNATIONAL SEARCH REPORT

International application No. PCT/US 10/53360

Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet) Box No. II This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons: Claims Nos.: because they relate to subject matter not required to be searched by this Authority, namely: Claims Nos.: because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically: Claims Nos.: because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a). Observations where unity of invention is lacking (Continuation of item 3 of first sheet) This International Searching Authority found multiple inventions in this international application, as follows: ----- Please see Extra Sheet -----As all required additional search fees were timely paid by the applicant, this international search report covers all searchable As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees. As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.: No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.: **Remark on Protest** The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee. The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation. No protest accompanied the payment of additional search fees.

Form PCT/ISA/210 (continuation of first sheet (2)) (July 2009)

#### INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No. PCT/US 10/53360

Box III Observations where unity of invention is lacking:

This application contains the following inventions or groups of inventions which are not so linked as to form a single general inventive concept under PCT Rule 13.1. In order for all inventions to be examined, the appropriate additional examination fees must be paid.

Group II: claims 1-83 Group II: claims 84-141 Group III: claims 142-185

Group I: Claims 1-83 are directed to polyelectrolyte hair care compositions comprising cationic polymers, anionic polymers, and other excipients and carriers; and also related methods of use.

Group II: Claims 84-141 are directed to methods of producing polyelectrolyte hair care compositions comprising cationic polymers, anionic polymers, and other excipients and carriers.

Group III: Claims 142-185 are directed to methods for quantitatively assessing the degree of hair repair resulting from the application of a polyelectrolyte hair care composition comprising cationic polymers, anionic polymers, and other excipients and carriers.

The inventions listed as Groups I-III do not relate to a single general inventive concept under PCT Rule 13.1 because, under PCT Rule 13.2, they lack the same or corresponding special technical features for the following reasons:

The groups share the technical feature of polyelectrolyte hair care compositions comprising cationic polymers, anionic polymers, and other excipients and carriers. However, these shared technical features fail to make a contribution over the prior art of US 2006/0251603 A1 (Rigoletto et al.) 09 November 2006 (09.11.2006), which discloses polyelectrolyte hair care compositions comprising cationic polymers, anionic polymers, and other excipients and carriers; as well as related methods of production and use. Accordingly, unity of invention is lacking under PCT Rule 13.1.

Form PCT/ISA/210 (patent family annex) (July 2009)