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- as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))
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(54) Title: HAIR TREATMENT COMPOSITION

(57) **Abstract**: The invention provides a hair treatment composition for the provision of multilayer benefits to the hair fibre, the composition comprising one or more silk sericins dissolved or dispersed in a gelled aqueous continuous phase comprising an anionic polymeric gelling agent selected from carboxylic acid polymers. The composition of the invention provides multilayer benefits to the hair fibre such as fibre strengthening, damage repair, hair shape smoothing and frizz control.



HAIR TREATMENT COMPOSITION

Field of the Invention

- 5 This invention relates to a hair treatment composition, and more particularly a hair treatment composition for the provision of multilayer benefits to the hair fibre.

Background of the Invention

- 10 The purpose of bleaching is to eliminate or lighten the natural hair colour by the reaction of an oxidizing agent with the melanin pigment. Examples of oxidizing agents that can be used are hydrogen peroxide, potassium, sodium or ammonium salts of perborate, percarbonate, persulfate and percarbamide, and mixtures thereof. Bleaches are also used during oxidative dyeing treatments. Oxidative (or "permanent") dye compositions comprise "precursor dyes" which are small molecules capable of
- 15 diffusing into the hair. These molecules mainly belong to three classes of aromatic compounds: diamines, aminophenols and phenols. They are sufficiently small to diffuse in the hair shaft where, once activated by an oxidizing agent such as hydrogen peroxide, they further react with other precursors to form larger coloured complexes.
- 20 Oxidative treatments of hair are very popular with consumers since they provide good results which are relatively unaffected by light, shampooing and perspiration. However the process is not without drawbacks. Repeated oxidative treatments over prolonged periods may damage or weaken hair, making it prone to breakage and reduced lustre.
- 25 Since the hair fibre is a multilayer structure, once hair is damaged, treatments must intervene on several levels to treat it - from the middle of the core to the surface of the cuticle.
- 30 Film-forming polymers are often used in treatments for damaged hair because they alter hair surface properties, imparting smoothing and gliding effects and shine, and have a significant impact on the macroscopic behavior of the hair array. However, film-forming polymers are by nature designed to provide hair fibres with a hydrophobic coating that may slow or prevent the penetration of actives. Therefore such treatments

may not provide multilayer benefits to the hair fibre such as fibre strengthening, damage repair, hair shape smoothing and frizz control.

The present invention addresses this problem.

5

Summary of the Invention

The present invention provides a hair treatment composition according to Claim 1.

Detailed Description and Preferred Embodiments

10 All molecular weights as used herein are weight average molecular weights, unless otherwise specified.

The hair treatment composition of the invention comprises *inter alia* a gelled aqueous continuous phase.

15

By “aqueous continuous phase” is meant a continuous phase which has water as its basis.

A hair treatment composition according to the invention will generally comprise at least
20 60%, preferably at least 70% and more preferably at least 80% water (by weight based on the total weight of the composition). Preferably, the composition comprises no more than 99% and more preferably no more than 98% water (by weight based on the total weight of the composition). Other organic solvents may also be present, such as lower alkyl alcohols and polyhydric alcohols. Examples of lower alkyl alcohols include C₁ to C₆
25 monohydric alcohols such as ethanol and isopropanol. Examples of polyhydric alcohols include propylene glycol, hexylene glycol, glycerin, and propanediol. Mixtures of any of the above-described organic solvents may also be used.

The hair treatment composition of the invention comprises *inter alia* one or more silk
30 sericins.

Silks can be broadly defined as externally spun fibrous protein secretions, made by arthropods for a variety of task-specific applications. Silk fibres are typically composite

materials formed of silk protein and other associated molecules such as glycoproteins and lipids.

Silkworms produce silk cocoons to protect themselves during their metamorphosis into
5 moths, and humans have harvested silk fibres from these cocoons for centuries to produce textiles. Of all natural silk-producing animals, mulberry silkworms (*Bombyx mori*) are of the most economic importance, because it is possible to rear them in captivity. Other than the domesticated *B.mori*, silk fibre production is reported from the wild non-mulberry saturniid variety of silkworms, such as tasar (*Antheraea mylitta*),
10 muga (*Antheraea assamensis*) and eri (*Philosamia ricini*).

Silkworm (e.g. *B.mori*) silks are composed of two groups of proteins. The fibroins, which constitute the silk thread, are synthesized in the posterior part of the silk gland (PSG). The sericins are produced by the middle silk gland (MSG), and are a family of
15 globular, water-soluble proteins, which ensure the cohesion of the cocoon by sticking the fibroin fibres together. The sericins are characterized by their high serine content, where serine represents at least about 20 mol% of the total amino acid residues. Typically serine represents from about 20 to about 40 mol%, preferably from about 30 to about 40 mol% of the total amino acid residues.

20 A peptide consisting of 38 amino acids has been identified as a highly conserved and internally repetitive sequence of *B.mori* silk sericins. The consensus sequence of this peptide (Ser-Ser-Thr-Gly-Ser-Ser-Ser-Asn-Thr-Asp-Ser-Asn-Ser-Asn-Ser-Ala-Gly-Ser-Ser-Thr-Ser-Gly-Gly-Ser-Ser-Thr-Tyr-Gly-Tyr-Ser-Ser-Asn-Ser-Arg-Asp-Gly-Ser-Val) is
25 characterized by its similarity to the average amino acid composition of silk sericin and its high hydrophilic amino acid content.

Native *B.mori* silk sericins range in molecular weight from about 10 to about 400 kDa, depending on gene coding and post-translational modifications. Three major fractions
30 of silk sericin have been isolated from the *B.mori* cocoon, with molecular weights of about 150, 250, and 400 kDa respectively.

Silk sericins for use in the invention may be naturally derived, typically by extraction from silkworm (e.g. *B.mori*) cocoons or by extraction from raw silk. During the extraction process, the silk sericin may be hydrolysed to a certain extent, depending on the extraction method, temperature, pH and processing time. Accordingly, the
5 molecular weight which is quoted for such materials will represent the average molecular weight of the various protein, polypeptide, oligopeptide and amino acid constituents present. The average molecular weight of silk sericins derived by boiling water extraction of cocoons generally ranges from about 65 kDa to about 400 kDa. If an alkaline solution is used, such as one containing sodium hydroxide, the average
10 molecular weight of the derived silk sericin generally ranges from about 1 kDa to about 50 kDa.

Silk sericins for use in the invention may also be artificially synthesized using conventionally known biological methods, for example, by inserting the silk sericin gene
15 sequence into *E. coli*, in which the *E. coli* produces recombinant silk sericin, or by conventionally known chemical methods such as Fmoc/tBu solid-phase peptide synthesis.

Artificially synthesized silk sericins will generally contain several repeats of the 38
20 amino acid consensus sequence described above (Ser-Ser-Thr-Gly-Ser-Ser-Ser-Asn-Thr-Asp-Ser-Asn-Ser-Asn-Ser-Ala-Gly-Ser-Ser-Thr-Ser-Gly-Gly-Ser-Ser-Thr-Tyr-Gly-Tyr-Ser-Ser-Asn-Ser-Arg-Asp-Gly-Ser-Val), for example 2 to 8 repeats, more preferably 2 to 6 repeats.

25 Mixtures of any of the above-described types of silk sericins may also be used in the invention.

Preferred silk sericins for use in the invention are naturally derived, and have an average molecular weight ranging from about 1 kDa to about 200 kDa, more preferably
30 from about 5 to about 100 kDa and most preferably from about 10 kDa to about 80 kDa,

An example of a preferred silk sericin for use in the invention is silk sericin extracted from silkworm (e.g. *B.mori*) cocoons or raw silk, and containing serine at a level of from about 30 to about 40 mol% of the total amino acid residues, and having an average molecular weight ranging from about 1 kDa to about 200 kDa, more preferably from about 5 to about 100 kDa and most preferably from about 10 kDa to about 80 kDa.

The hair treatment composition of the invention has been found to be effective for the provision of multilayer benefits to the hair fibre, even when it comprises relatively low levels of dissolved or dispersed silk sericin(s). Accordingly, in a typical hair treatment composition according to the invention, the level of silk sericin(s) ranges from 0.3 to 0.7% (by weight based on the total weight of the composition).

The hair treatment composition of the invention comprises *inter alia* an anionic polymeric gelling agent selected from carboxylic acid polymers.

The term "carboxylic acid polymer" in the context of this invention generally denotes a homopolymer or copolymer obtained from the polymerization of ethylenically unsaturated monomers containing pendant carboxylic acid groups (hereinafter termed "carboxylic monomers").

Suitable carboxylic monomers generally have one or two carboxylic acid groups, one carbon to carbon double bond and contain a total of from 3 to about 10 carbon atoms, more preferably from 3 to about 5 carbon atoms.

Specific examples of suitable carboxylic monomers include α - β -unsaturated monocarboxylic acids such as acrylic acid, methacrylic acid and crotonic acid; and α - β -unsaturated dicarboxylic acids such as itaconic acid, fumaric acid, maleic acid and aconitic acid. Salts, esters or anhydrides of the α - β -unsaturated mono- or dicarboxylic acids described above may also be used. Examples include half esters of α - β -unsaturated dicarboxylic acids with C1-4 alkanols, such as monomethyl fumarate; cyclic anhydrides of α - β -unsaturated dicarboxylic acids such as maleic anhydride, itaconic anhydride and citraconic anhydride; and esters of acrylic acid or methacrylic

acid with C1-30 alkanols, such as ethyl acrylate, butyl acrylate, 2-ethylhexyl acrylate, dodecyl acrylate, hexadecyl acrylate, and octadecyl acrylate.

5 Optionally, other ethylenically unsaturated monomers can be copolymerized into the carboxylic acid polymer backbone. Example of such other ethylenically unsaturated monomers include styrene, vinyl acetate, ethylene, butadiene, acrylonitrile and mixtures thereof.

10 Carboxylic acid polymers for use as the anionic polymeric gelling agent in the invention preferably have a molecular weight of at least 1 million g/mol.

Carboxylic acid polymers for use as the anionic polymeric gelling agent in the invention may suitably be crosslinked.

15 Typical crosslinking monomers include polyalkenyl polyethers having at least two polymerizable ethylenically unsaturated double bonds. The term "polyalkenyl polyether" in the context of this invention refers to alkenyl ethers of organic polyols wherein the organic polyol is etherified by reacting it with an alkenyl halide, such as allyl chloride or allyl bromide. The polyalkenyl polyether (e.g. polyallyl polyether) can contain 2 to 8
20 polymerizable ethylenically unsaturated double bonds. Suitable polyols for the etherification reaction can contain 2 to 12 carbon atoms and have at least two hydroxyl groups. The polyol can be linear, branched or cyclic (e.g. monosaccharides and polysaccharides containing 1 to 4 saccharide units). Specific examples of suitable polyalkenyl polyethers include polyallyl ethers of sucrose having from 2 to 8 allyl
25 groups per molecule, pentaerythritol diallyl ether, pentaerythritol triallyl ether, pentaerythritol tetraallyl ether; trimethylolpropane diallyl ether, trimethylolpropane triallyl ether, and mixtures thereof.

A suitable carboxylic acid polymer for use as the anionic polymeric gelling agent in the
30 invention is a crosslinked homopolymer polymerized from acrylic acid or methacrylic acid (typically crosslinked with an allyl ether of pentaerythritol, or an allyl ether of sucrose). Such materials may generally be referred to under the INCI name of

Carbomer. Commercially available examples include CARBOPOL® polymers 934, 940 and 980 and CARBOPOL® Ultrez 10 from Lubrizol Advanced Materials.

Also suitable are crosslinked copolymers polymerized from C1-4 alkyl acrylate or methacrylate (e.g. ethyl acrylate) with one or more comonomers selected from acrylic acid, methacrylic acid and mixtures thereof (typically crosslinked with an allyl ether of pentaerythritol). Such materials may generally be referred to under the INCI name of Acrylates Copolymer. Commercially available examples include ACULYN® 33 from Rohm and Haas.

10

Also suitable are crosslinked copolymers polymerized from C10-30 alkyl esters of acrylic or methacrylic acid with one or more comonomers selected from acrylic acid, methacrylic acid and their respective C1-4 alkyl esters (typically crosslinked with an allyl ether of pentaerythritol). Such materials may generally be referred to under the INCI name of Acrylates/C10-30 Alkyl Acrylate Crosspolymer. Commercially available examples include CARBOPOL® polymers 1342 and 1382 from Lubrizol Advanced Materials.

15

Also suitable are optionally crosslinked copolymers of acrylic acid or methacrylic acid with alkyl acrylates and ethoxylated hydrophobically modified alkyl acrylates. Such materials may generally be referred to under the INCI names of Acrylates/Steareth-20 Methacrylate Copolymer, Acrylates/Beheneth-25 Methacrylate Copolymer, Acrylates/Steareth-20 Methacrylate Crosspolymer and Acrylates/Palmeth-25 Acrylates Copolymer. Commercially available examples include ACULYN® 22, 28 or 88 from Rohm & Haas and SYNTHALEN® from 3V Sigma.

20

25

Carboxylic acid polymers for use as the anionic polymeric gelling agent in the invention are preferably selected from Carbomers, such as homopolymers of acrylic acid crosslinked with an allyl ether of pentaerythritol or an allyl ether of sucrose.

30

Mixtures of any of the above-described materials may also be used.

In a typical composition according to the invention, the level of carboxylic acid polymers ranges from 0.2 to 0.6% by weight based on the total weight of the composition.

5 In a particularly preferred composition according to the invention the carboxylic acid polymer is one or more homopolymers of acrylic acid crosslinked with an allyl ether of pentaerythritol or an allyl ether of sucrose; at a level ranging from 0.4 to 0.8% by weight based on the total weight of the composition.

10 In formulations containing anionic polymeric gelling agents such as the carboxylic acid polymers described above, it is often necessary to neutralize at least a portion of the free carboxyl groups by the addition of an inorganic or organic base. Examples of suitable inorganic or organic bases include alkali metal hydroxides (e.g. sodium or potassium hydroxide), sodium carbonate, ammonium hydroxide, methylamine, diethylamine, trimethylamine, monoethanolamine, triethanolamine and mixtures
15 thereof.

The pH of the final, fully-formulated composition of the invention preferably ranges from 4 to 7, more preferably from 5.5 to 6.5.

20 The viscosity of the composition according to the invention will generally range from about 5,000 to about 50,000 mPa.s and preferably ranges from 10,000 to 20,000 mPa.s (Brookfield RVT, 20 rpm @ 25°C).

25 A hair treatment composition according to the invention may also incorporate other optional ingredients to enhance performance and/or consumer acceptability. Suitable optional ingredients include preservatives, colouring agents, chelating agents, antioxidants, fragrances, antimicrobials and sunscreens.

30 The composition of this invention is suitable for topical application to hair to improve hair volume-down. The term "volume-down" in the context of this invention generally means reduced visible bulkiness of the hair. For many consumers, improved hair volume-down provides a number of associated benefits, such as improved straightness, manageability and style retention.

The composition of this invention is also suitable for topical application to damaged hair, such as oxidatively treated hair. The composition is especially suitable for treating oxidatively damaged hair to provide benefits to the hair fibre such as fibre strengthening, damage repair, hair shape smoothing and frizz control.

5

As used herein, the term "oxidatively-treated hair" means hair that has been subjected to any treatment comprising at least one step of contacting the hair with at least one oxidizing composition. Examples of oxidative treatments for human hair are bleaching, dyeing or perming.

10

As used herein, the term "oxidizing composition" means a composition comprising at least one oxidizing agent suitable for use on hair, such as hydrogen peroxide, potassium, sodium or ammonium salts of perborate, percarbonate, persulfate and percarbamide, and mixtures thereof. Examples of such compositions are oxidative dye compositions and bleaching compositions.

15

In a typical method of treating hair with the composition of the invention, the composition is topically applied to the hair and then worked into the hair. Preferably the composition is applied to the hair in a single dose, and at a dosage level of from 0.01 to 0.5g, more preferably from 0.02 to 0.2g and most preferably from 0.05 to 0.15g of composition per gram of hair.

20

Preferably the composition is applied as described above to damp hair.

Preferably the hair is dried or allowed to dry after application of the composition as described above, without rinsing the composition from the hair. The hair may be allowed to dry naturally by exposure to air, or may be dried by use of a heated hair drying appliance, by rubbing with a water-absorbent article, or by a combination of any of these methods.

30

The dried hair may then be mechanically shaped if required.

The composition may then be rinsed from the hair, suitably at the next wash, which will typically be after a period of about 24 to 72 hours following the initial application of the composition.

- 5 Mechanical shaping of the hair can be accomplished by such means as the fingertips, a plastic hair pick or the tail of a comb, the shaping being performed on portions of the hair comprising strands of hair in various numbers. Using such means the hair may be pulled, combed, smoothed, pressed or flattened into a straightened configuration; or shaped gently into bends, waves or curls.

10

Preferably the hair is mechanically shaped by mechanically straightening it. For example, the hair may be pulled, combed, smoothed, pressed or flattened into a straightened configuration.

- 15 A hot tool, such as an electrically heated flat hair iron or hand-held hair dryer, may be used in the mechanical shaping step. Such tools apply high levels of heat directly to the hair. Most operate in the 45°C to 250°C range, and are usually employed at temperature settings ranging from 50°C to about 220°C, depending on the particular tool.

20

However, the use of hot tools is not essential in the mechanical shaping step. This is especially advantageous for consumers who wish to reduce or avoid the exposure of their hair to high temperatures, for example if their hair is fragile or overprocessed from previous chemical treatments such as bleaching and perming.

25

Accordingly the mechanical shaping of the hair is preferably conducted at a temperature from 15 to 40°C, such as from 20 to 30°C.

- 30 Preferably the hair is mechanically shaped by combing it into a straightened configuration at a temperature from 15 to 40°C, such as from 20 to 30°C.

The invention is further illustrated with reference to the following, non-limiting Examples.

EXAMPLES

In the Examples, all ingredients are expressed by weight percent of the total formulation, and as level of active ingredient.

- 5 Gelled aqueous hair treatment formulations were prepared, having ingredients as shown in Table 1. Comparative Examples (not according to the invention) are indicated by letter. A number indicates Examples according to the invention.

Table 1

Ingredient	Example A	Example 1	Example 2
	wt%		
Carbomer (CARBOPOL® Ultrez 10, ex Lubrizol Advanced Materials)	0.4	0.4	0.4
Methyl paraben	0.2	0.2	0.2
TEA	1	1	1
Water	98.4	97.9	97.9
Silk sericin powder molecular weight 70kDa (extracted at high temperature (> 110°C) in water at high pressure)	-	0.5	-
Silk sericin powder molecular weight 20 kDa (ex Huzhou Xintiansi Bio-tech Co., Ltd., China)	-	-	0.5

10

The formulations of Examples 1 and 2 were prepared by first dissolving the carbomer and methyl paraben in a portion of the water in a main vessel. In a separate vessel, the TEA was diluted using a portion of the water in a 1:1 ratio. The carbomer-methyl

15 paraben-water solution in the main vessel was then agitated using a Silverson mixer at 2500 rpm. The TEA solution was added dropwise to the main vessel with stirring. Finally the silk sericin was dissolved in the remainder of the water in a separate vessel and added to the main vessel. The resulting formulation was stirred for 5 more minutes and then stored at room temperature in a Tarson container.

20

The formulation of Example A was prepared as above, with the omission of the silk sericin.

Experiment 1

5

The formulations of Example A and Example 1 respectively were used to treat virgin dark brown European wavy #6 switches of length 25 cm and weight 2 grams.

10 In each case, the hair switch is lightly wetted, then a dose of 0.2 gram of test formulation is applied to the hair switch and worked into the hair switch. This gives a dosage level of 0.1 gram of test formulation per gram of hair.

The switches were combed straight and when dry they were combed and pictures taken. The volumes* of the switches were measured using an image analysis kit and
15 the results are shown in Table 2. The volume shows the volume-down (straightness) benefits of the treatment.

*(**"volume" refers to the projection of the switch image on to the screen and is given in mm²).*

20

Table 2

Treatment	Volume (mm ²)
Example A	13381
Example 1	10600

The results show that the switches treated with the formulation of Example 1 are much
25 straighter (volume-down) compared to the switches treated with the formulation of Example A. The difference was seen to be statistically significant (> 99%).

A similar shape benefit was seen when the switches were treated with the formulation of Example 2.

30

Experiment 2

The formulations of Example A and Example 1 respectively were used to treat twice-bleached dark brown European wavy#6 switches of length 25 cm and weight 2 grams.

5

In each case, a dose of 0.2 gram of test formulation is applied to the hair switch and worked into the hair switch. This gives a dosage level of 0.1 gram of test formulation per gram of hair.

10 A sample of fibres from each of the treated hair switches was removed and prepared for wet DSC measurements. The denaturation temperature Td was measured and the results are shown in Table 3.

Table 3

15

Treatment	Td (°C)
Example A	143.6
Example 1	144.6

The results show that the switches treated with the formulation of Example 1 have a much higher Td compared to the switches treated with the formulation of Example A. The difference was seen to be statistically significant (> 99%).

20

It can be seen from the results that the formulation of Example A (not according to the invention) provides a very marginal volume-down benefit and no fibre strengthening benefit. By contrast, the formulation of Example 1 (according to the invention) significantly improves the shape and fibre strengthening of hair. This is surprising
25 since a dose of 0.2 gram of test formulation on a 2 gram hair switch works out to a dosage on the hair of only 500 ppm of silk sericin.

CLAIMS

1. A hair treatment composition comprising one or more silk sericins dissolved or dispersed in a gelled aqueous continuous phase comprising an anionic polymeric gelling agent selected from carboxylic acid polymers, wherein the level of silk sericin(s) ranges from 0.3 to 0.7% (by weight based on the total weight of the composition) and wherein the level of carboxylic acid polymer (s) ranges from 0.2 to 0.6% (by weight based on the total weight of the composition).
2. A composition according to claim 1, in which the silk sericin (s) are extracted from *B.mori* cocoons or raw silk, and contain serine at a level of from 30 to 40 mol% of the total amino acid residues, and have an average molecular weight ranging from 1 kDa to 200 kDa.
3. A composition according to claim 1 or claim 2, in which the average molecular weight of the silk sericin (s) ranges from 10 kDa to 80 kDa.
4. A composition according to any one of claims 1 to 3, in which the carboxylic acid polymer is selected from homopolymers of acrylic acid crosslinked with an allyl ether of pentaerythritol or an allyl ether of sucrose.
5. A method of treating hair to provide benefits to the hair fibre, comprising the steps of topically applying to the hair a composition according to any one of claims 1 to 4, followed by drying the hair or allowing it to dry, without rinsing the composition from the hair.
6. A method according to claim 5, further comprising the step of mechanically shaping the dried hair by combing it into a straightened configuration at a temperature from 15 to 40°C.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2017/077957

A. CLASSIFICATION OF SUBJECT MATTER
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ADD.

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
A61Q A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2008 273907 A (DAI ICHI KOGYO SEIYAKU CO LTD) 13 November 2008 (2008-11-13) paragraph [0020]; examples 1-3; table 1 -----	1-6
A	Anonymous: "Sericin - Wikipedia", 7 November 2016 (2016-11-07), XP055327456, Retrieved from the Internet: URL:https://en.wikipedia.org/wiki/Sericin [retrieved on 2016-12-08] paragraphs [0001] - [0003] -----	1-6
A	JP 2007 015951 A (KANEBO HOME PRODUCTS KK) 25 January 2007 (2007-01-25) example 14 ----- -/-	1-6



Further documents are listed in the continuation of Box C.



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PCT/EP2017/077957

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
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