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(54) **Title:** HAIR CARE COMPOSITION

(57) **Abstract:** A hair colouring composition comprising an emulsion comprising: a) hydrophobically modified particles selected from the group consisting of silica, metal oxide and mixtures thereof; b) amino functionalised silicone; c) an iron salt or complex; and d) water.



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Hair Care Composition

The present invention relates to hair colouring compositions.

- 5 Polyphenol compound mordanted with metal ions have been known to dye human hair for many years. Colouring compositions containing metal salts are described in EP0327345 (Beecham) WO2010/135237, (Advance Cosmetic Technologies); WO/2007/130777 and WO 2000/29036 (Henkel).
- 10 However compositions comprising metal salts, such as iron, are frequently unstable on storage. The present invention mitigates the problem of instability, in particular phase separation, of colourant compositions comprising iron.

Summary of the Invention

15

Accordingly the present invention provides a hair colouring composition comprising an emulsion comprising:

- a) hydrophobically modified particles selected from the group consisting of silica, metal oxide and mixtures thereof;
- 20 b) amino functionalised silicone;
- c) an iron salt or complex; and
- d) water.

The invention further relates to a kit for colouring hair comprising:

- 25 (i) a first composition comprising a polyphenol or source of polyphenol
- (ii) a second composition as claimed in any preceding claim.

Also described is a method for colouring hair comprising the steps of applying to the hair a composition or kit described in above.

30

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Description of the Invention

Emulsion

- 5 Compositions of the invention may be water in oil or oil in water based emulsions. Preferably they are oil in water based emulsions.

Unless stated otherwise the viscosity of silicones can be measured by means of a glass capillary viscometer as set out further in Dow Corning Corporate Test Method
10 CTM004, July 20 1970.

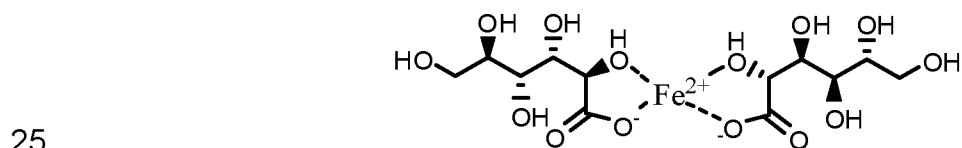
Iron Salt or Complex

Compositions of the invention comprise a iron salt or complex.
15

Iron containing compositions of the invention preferably comprise from 0.01wt% to 10 wt% of the total of iron composition of iron, more preferably from 0.1wt% to 5wt% of the total composition, more preferably from 0.2 to 4 wt%.

- 20 The level of metal ion in the formulation may be determined by quantitative elemental analysis.

For the avoidance of doubt, if the formulation contains 2wt% of complex



With molecular weight of 446.14, then it will contain $55.84/446.14 \times 2 = 0.25\text{wt\%}$ of Fe(II) (2 decimal places).

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The iron may be present as a salt or as a complex.

If iron is present as a salt it is preferred if the salt is iron chloride.

5

If present as a complex with a ligand it is preferable if the ligand is organic in nature.

10 In the context of this invention a ligand is a molecule that contains more than one group, preferably 2 to 4 groups, most preferably 2 groups that co-ordinate with the metal ion. Groups for co-ordinating with the metal ion are COO^- , OH, ketones, esters, primary amines, secondary amines and tertiary amines, more preferably COO^- , tertiary amines and OH, most preferably COO^- and OH. Polyphenols are not permitted as multidentate ligands.

15

The ligand-metal complex may be pre-formed before addition to the composition for example iron (II) gluconate, iron (II) glutamate. The ligand metal complex may be formed in the composition by the addition of an iron salt and the ligand, for example FeCl_2 and sodium lactate. Preferably, the mole ratio of iron ion to ligand
20 is from 1:1 to 1:4, more preferably from 1:1.5 to 1:2.5.

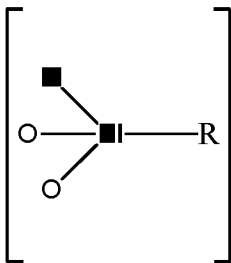
Preferably, the metal ion is in the form of a complex that is soluble in aqueous solution at the pH of the formulation. Most preferably the metal ion in the form of a salt and/or complex has a solubility in demineralised water at the pH of the
25 formulation of greater than 0.1g/L.

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Hydrophobically modified particles

The compositions of the invention comprise hydrophobically modified particles of silica or metal oxide. Preferably the hydrophobically particles comprise silica or
 5 titanium oxide, more preferably hydrophobically modified silica (silicone dioxide) .

It is particularly preferred if the hydrophobically modified silica conforming to the formula:

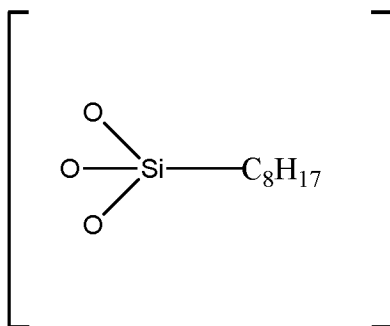


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in which R is a C₄ to C₁₈ alkyl group.

Particularly preferred is hydrophobically modified silicone dioxide conforming to the formula:

15



Preferred silica particles are described in US 7 282 236 and made commercially
 20 available from suppliers like Evonik Degussa GmbH under the names Aerosil R812, R202, and R805. Silica having a C₄ to C₁₅ alkyl group attached to the silane are

- 5 -

preferred. Particularly preferred is octylsilane comprising the group represented by formula above (sold under the name Aerosil R805).

5 The hydrophobically modified silica preferably has a primary particle size (in its dry state) from 1nm to 100 nm, more preferably from 5 to 70nm.

Primary particles sizes can be derived from Transmission Electron Microscopy according to the method described by S. Gu et al in Journal of Colloid and Interface Science 289 (2005) 419-426.

10

Composition of the invention preferably comprise from 0.05 to 15 wt% of the total composition of hydrophobically modified silica, more preferably from 0.1 to 10 wt%, and most preferably from 0.2 to 5 wt%

15 **Amino Functionalised Silicone**

Compositions according to the invention comprise an amino functional silicone. By "amino functionalised silicone" is meant a silicone containing at least one primary, secondary or tertiary amine group, or a quaternary ammonium group.

20

The primary, secondary, tertiary and/or quaternary amine groups may either form part of the main polymer chain or more preferably be carried by a side or pendant group carried by the polymeric backbone.

25 Suitable amino functionalised silicone polymers for use with the invention are described in US 4 185 087.

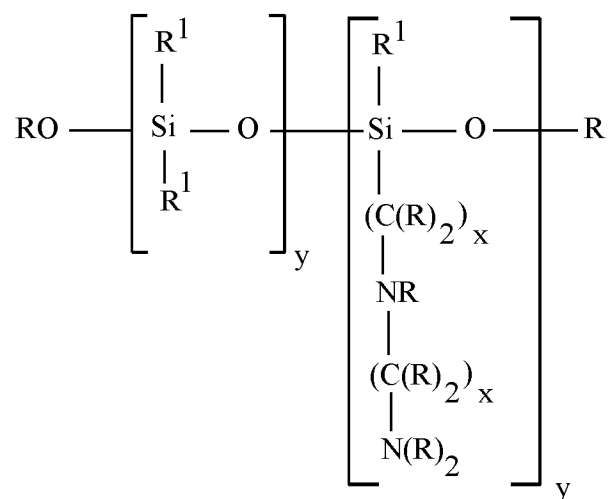
Amino functionalised silicones suitable for use in the invention will typically have a mole % amine functionality in the range of from about 0.1 to about 8.0 mole %, preferably from about 0.1 to about 5.0 mole %, most preferably from about 0.1 to

30

- 6 -

about 2.0 mole %. In general the amine concentration should not exceed about 8.0 mole % since we have found that too high an amine concentration can be detrimental to total silicone deposition and therefore conditioning performance.

- 5 In a preferred embodiment, the functionalized polymers are of the amodimethicone having the general formula:



- 10 where each R is independently H, or a C₁₋₄ alkyl, preferably H;
 each R¹ is independently OR or a C₁₋₄ alkyl; and
 each x is independently an integer from 1 to 4 and each y is greater than zero and
 independently an integer to yield a polymer having a molecular weight from 500 to 1
 15 million, and preferably, from 750 to 25,000, and most preferably from 1,000 to
 15,000. Particularly preferred are silicones comprising an amino glycol copolymer.
 Especially preferred is (C13-C15 alkoxy) PG amodimethicone such as DC 8500 by
 Dow Corning .

- It is also within the scope of this invention for the functionalized polymer to comprise
 20 trimethylsilylamodimethicone.

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Preferably the level of amino functionalised silicone polymers make up from 0.05 to 15 wt% of the total composition, preferably from 0.1 to 10 wt%, and most preferably from 0.2 to 5 wt%.

- 5 Preferably the weight ratio of hydrophobically modified particle to amino functionalised silicone is from 3:1 to 1:50, more preferably from 1:1 to 1:10.

When a high ratio (greater than 1:1) of hydrophobically modified particle to amino silicone is used it is preferred if the a non-amino functional silicone is present,
10 preferably at a weight ratio of greater than 1:1 non-amino functional silicone to amino functional silicone, more preferably at a ratio of greater than 2:1.

Suitable non-amino functional silicones are described in the paragraphs below.

15 **Conditioning Phase**

Preferably the iron containing emulsion is present in a conditioner base.

Such conditioner bases will typically comprise one or more conditioning surfactants
20 which are cosmetically acceptable and suitable for topical application to the hair.

Suitable conditioning surfactants include those selected from cationic surfactants, used singly or in admixture. Preferably, the cationic surfactants have the formula $N^+R^1R^2R^3R^4$ wherein R^1 , R^2 , R^3 and R^4 are independently (C_1 to C_{30}) alkyl or
25 benzyl. Preferably, one, two or three of R^1 , R^2 , R^3 and R^4 are independently (C_4 to C_{30}) alkyl and the other R^1 , R^2 , R^3 and R^4 group or groups are (C_1 - C_6) alkyl or benzyl. More preferably, one or two of R^1 , R^2 , R^3 and R^4 are independently (C_6 to C_{30}) alkyl and the other R^1 , R^2 , R^3 and R^4 groups are (C_1 - C_6) alkyl or benzyl groups. Optionally, the alkyl groups may comprise one or more ester (-OCO- or -
30 COO-) and/or ether (-O-) linkages within the alkyl chain. Alkyl groups may

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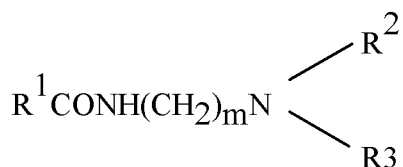
optionally be substituted with one or more hydroxyl groups. Alkyl groups may be straight chain or branched and, for alkyl groups having 3 or more carbon atoms, cyclic. The alkyl groups may be saturated or may contain one or more carbon-carbon double bonds (eg, oleyl). Alkyl groups are optionally ethoxylated on the alkyl chain with one or more ethyleneoxy groups.

Suitable cationic surfactants for use in conditioner compositions according to the invention include cetyltrimethylammonium chloride, behenyltrimethylammonium chloride, cetylpyridinium chloride, tetramethylammonium chloride, tetraethylammonium chloride, octyltrimethylammonium chloride, dodecyltrimethylammonium chloride, hexadecyltrimethylammonium chloride, octyldimethylbenzylammonium chloride, decyldimethylbenzylammonium chloride, stearyldimethylbenzylammonium chloride, didodecyldimethylammonium chloride, dioctadecyldimethylammonium chloride, tallowtrimethylammonium chloride, dihydrogenated tallow dimethyl ammonium chloride (eg, Arquad 2HT/75 from Akzo Nobel), cocotrimethylammonium chloride, PEG-2-oleammonium chloride and the corresponding hydroxides thereof. Further suitable cationic surfactants include those materials having the CTFA designations Quaternium-5, Quaternium-31 and Quaternium-18. Mixtures of any of the foregoing materials may also be suitable. A particularly useful cationic surfactant for use in conditioners according to the invention is cetyltrimethylammonium chloride, available commercially, for example as GENAMIN CTAC, ex Hoechst Celanese. Another particularly useful cationic surfactant for use in conditioners according to the invention is behenyltrimethylammonium chloride, available commercially, for example as GENAMIN KDMP, ex Clariant.

Another example of a class of suitable cationic surfactants for use in the invention, either alone or together with one or more other cationic surfactants, is a combination of (i) and (ii) below:

(i) an amidoamine corresponding to the general formula (I):

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in which R^1 is a hydrocarbyl chain having 10 or more carbon atoms,
 R^2 and R^3 are independently selected from hydrocarbyl chains of from 1 to 10
 5 carbon atoms, and
 m is an integer from 1 to about 10; and

(ii) an acid.

10 As used herein, the term hydrocarbyl chain means an alkyl or alkenyl chain.

Preferred amidoamine compounds are those corresponding to formula (I) in which

R^1 is a hydrocarbyl residue having from about 11 to about 24 carbon atoms,
 15 R^2 and R^3 are each independently hydrocarbyl residues, preferably alkyl groups,
 having from 1 to about 4 carbon atoms, and m is an integer from 1 to about 4.

Preferably, R^2 and R^3 are methyl or ethyl groups.

20 Preferably, m is 2 or 3, i.e. an ethylene or propylene group.

Preferred amidoamines useful herein include stearamido-propyldimethylamine,
 stearamidopropyldiethylamine, stearamidoethyldiethylamine,
 stearamidoethyldimethylamine, palmitamidopropyldimethylamine,
 25 palmitamidopropyl-diethylamine, palmitamidoethyldiethylamine,
 palmitamidoethyldimethylamine, behenamidopropyldimethyl-amine,
 behenamidopropyldiethylamine, behenamidoethyldiethyl-amine,

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behenamidoethyldimethylamine, arachidamidopropyl-dimethylamine, arachidamidopropyldiethylamine, arachid-amidoethyldiethylamine, arachidamidoethyldimethylamine, and mixtures thereof.

- 5 Particularly preferred amidoamines useful herein are stearamidopropyldimethylamine, stearamidoethyldiethylamine, and mixtures thereof.

Commercially available amidoamines useful herein include:

- 10 stearamidopropyldimethylamine with tradenames LEXAMINE S-13 available from Inolex (Philadelphia Pennsylvania, USA) and AMIDOAMINE MSP available from Nikko (Tokyo, Japan), stearamidoethyldiethylamine with a tradename AMIDOAMINE S available from Nikko, behenamidopropyldimethylamine with a tradename INCROMINE BB available from Croda (North Humberside, England),
15 and various amidoamines with tradenames SCHERCODINE series available from Scher (Clifton New Jersey, USA).

A protonating acid may be present. Acid may be any organic or mineral acid which is capable of protonating the amidoamine in the conditioner composition.

- 20 Suitable acids useful herein include hydrochloric acid, acetic acid, tartaric acid, fumaric acid, lactic acid, malic acid, succinic acid, and mixtures thereof. Preferably, the acid is selected from the group consisting of acetic acid, tartaric acid, hydrochloric acid, fumaric acid, lactic acid and mixtures thereof.

- 25 The primary role of the acid is to protonate the amidoamine in the hair treatment composition thus forming a tertiary amine salt (TAS) in situ in the hair treatment composition. The TAS in effect is a non-permanent quaternary ammonium or pseudo-quaternary ammonium cationic surfactant.

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Suitably, the acid is included in a sufficient amount to protonate more than 95 mole% (293 K) of the amidoamine present.

5 In conditioners of the invention, the level of cationic surfactant will generally range from 0.01 % to 10%, more preferably 0.05 % to 7.5%, most preferably 0.1 % to 5% by weight of the composition.

Conditioners of the invention will typically also incorporate a fatty alcohol. The combined use of fatty alcohols and cationic surfactants in conditioning compositions
10 is believed to be especially advantageous, because this leads to the formation of a lamellar phase, in which the cationic surfactant is dispersed.

Representative fatty alcohols comprise from 8 to 22 carbon atoms, more preferably 16 to 22. Fatty alcohols are typically compounds containing straight chain alkyl
15 groups. Examples of suitable fatty alcohols include cetyl alcohol, stearyl alcohol and mixtures thereof. The use of these materials is also advantageous in that they contribute to the overall conditioning properties of compositions of the invention.

The level of fatty alcohol in conditioners of the invention will generally range from
20 0.01 % to 10%, preferably from 0.1 % to 8%, more preferably from 0.2 % to 7%, most preferably from 0.3% to 6% by weight of the composition. The weight ratio of cationic surfactant to fatty alcohol is suitably from 1:1 to 1:10, preferably from 1:1.5 to 1:8, optimally from 1:2 to 1:5. If the weight ratio of cationic surfactant to fatty alcohol is too high, this can lead to eye irritancy from the composition. If it is too low,
25 it can make the hair feel squeaky for some consumers.

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

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the CTFA designation dimethiconol. Also suitable for use in compositions of the invention are silicone gums having a slight degree of cross-linking, as are described for example in WO 96/31188.

- 5 The viscosity of the emulsified silicone itself (not the emulsion or the final hair conditioning composition) is typically at least 10,000 cst. The viscosity of the silicone itself is preferably at least 60,000 cst, most preferably at least 500,000 cst, ideally at least 1,000,000 cst. Preferably the viscosity does not exceed 10^9 cst for ease of formulation.

10

Emulsified silicones for use the invention will typically have an average silicone particle size in the composition of less than 30, preferably less than 20, more preferably less than 10 microns. Most preferably the average silicone particle size of the emulsified silicone in the composition is less than 2 microns, ideally it ranges from 0.01 to 1 micron. Silicone emulsions having an average silicone particle size of ≤ 0.15 microns are generally termed microemulsions.

15

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

20

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

Examples of suitable pre-formed emulsions include emulsions DC2-1766,

25

DC2-1784, and microemulsions DC2-1865 and DC2-1870, all available from Dow Corning. These are all emulsions/microemulsions of dimethiconol. Cross-linked silicone gums are also available in a pre-emulsified form, which is advantageous for ease of formulation. A preferred example is the material available from Dow Corning as DC X2-1787, which is an emulsion of cross-linked dimethiconol gum.

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A further preferred example is the material available from Dow Corning as DC X2-1391, which is a microemulsion of cross-linked dimethiconol gum.

5 The total amount of silicone incorporated into compositions of the invention depends on the level of conditioning desired and the material used. A preferred amount is from 0.01 to about 15% by weight of the total composition.

Product form

10 To avoid oxidation of the metal by air it is preferred that the composition is stored in an air tight container such as a bottle closed with an air tight cap.

Colouring Kit

15 In a preferred embodiment of the invention the iron containing composition is sold as part of a kit together with a second composition comprising a polyphenol.

Preferably the polyphenol is selected from gallic acid, methyl gallate, ethyl gallate propyl gallate or mixtures thereof.

20

Methyl gallate, ethyl gallate, propyl gallate are obtainable by esterification of gallic acid. Preferably the gallic acid is extracted from a natural source, preferably from hydrolysable tannins. Preferably, the polyphenols do not contain any active oxidase enzymes and have been heat treated to destroy any enzyme activity.

25

The polyphenol is preferably selected from methyl gallate, ethyl gallate and propyl gallate, most preferably propyl gallate.

30 The polyphenol containing composition is preferably an aqueous polyphenol solution. The polyphenol composition preferably comprises from preferably

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comprises from 0.05 wt% to 10.0 wt% of the total polyphenol composition, more preferably from 0.1 wt% to 5.0 wt %, most preferably 0.3 wt% to 3.0 wt% of polyphenol.

- 5 Wherein the aqueous polyphenol solution preferably has a pH of 2 to 7 measured using a calibrated pH meter more preferably from 3 to 6.

To avoid oxidation of the polyphenols by air it is preferred that the composition is stored in an airtight container, preferably a bottle closed with an air tight cap.

10

The aqueous polyphenol solution contains water, preferably as the dominant ingredient. Auxiliary ingredients may be present for example to increase the viscosity, perfume and help solubilise the polyphenol. Solubilising ingredients include organic solvents and surfactants.

15

Product Form

Compositions of the invention are typically "rinse-off" compositions to be applied to the hair and then rinsed away.

20

Other Optional Ingredients

A composition of the invention may contain other ingredients for enhancing performance and/or consumer acceptability. Such ingredients include fragrance, dyes and pigments, pH adjusting agents, pearlescers or opacifiers, viscosity
25 modifiers, preservatives, and natural hair nutrients such as botanicals, fruit extracts, sugar derivatives and amino acids.

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Method of use

Composition of the invention are for colouring the hair, compositions are applied to the hair preferably followed by rinsing the hair with water.

5

Depending on the product type the time left on the hair can vary, products designed for a single colouring step are usually left on the hair for 5 to 30 minutes.

Compositions used for progressive colouring are left on the hair for 10 seconds to 10 minutes.

10

The method of colouring hair preferably comprises the steps of applying to hair sequentially in any order:

(i) the iron containing composition described above

15

(ii) a second composition comprising polyphenol.

Preferably, the compositions of the invention are applied to wet hair, necessitating the step of wetting the hair before application of the compositions of the invention.

A method preferably comprises the step of rinsing hair between application of the iron containing composition and the polyphenol composition. It is highly preferred if the hair is rinsed after application of both compositions of the invention.

20

Further conditioning and/or styling products may be applied as part of the colouring process.

25

The level of each composition applied to the head of hair is preferably from 5g to 100g.

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Preferably, the water used to wet and rinse the hair has a French hardness of from 0 to 36 degrees, more preferably 0 to 24 degrees, most preferably from 0 to 2 degrees.

- 5 Preferably, the water used to wet and rinse the hair contains less than 1ppm of chlorine based bleaching agents such as chlorine dioxide or hypochlorite. Most preferably less than 50ppb

The invention will now be illustrated by the following non-limiting examples.

- 10 Examples of the invention are illustrated by a number comparative examples are illustrated by a letter.

Examples

15 **Formulation Process**

- To form compositions of the invention the silica particles are dispersed in the silicone oils (aminosilicone oil DC8500/Dimethicone 5cst). Metal salts, and some of the water are added to the oil phase by stirring to form an emulsion. The
20 remainder of the cream is made in the conventional manner by mixing the ingredients, the emulsion is added to the cream.

Comparative Examples are made in the conventional manner by mixing the ingredients together.

25

The process takes place under ambient temperature.

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Table 1

			Example A	Example B	Example 1
Trade name	INCI name	Supplier	Activity (%) in Formula	Activity (%) in Formula	Activity (%) in Formula
Genamin BTLF	Behenyltrimonium Chloride	Clariant	1.61	1.61	1.288
Lexamine s-13	Steramidopropyl dimethylamine	Inolex	0.38	0.38	0.3
Lanette S3	Cetearyl alcohol	Ecogreen Oleo chemicals	4.00	4.00	3.2
DC 5-7134 9:1 600K/8566 CTAC 70%	Dimethicone/amodimethicone/Cetrimonium Chloride	Dow corning	3.50	3.50	2.8
Iron(III) chloride	Iron(III) chloride	Sigma-Aldrich	0.00	4.00	4.00
Aerosil R805	Hydrophobic silica	Dow corning	0.00	0.00	1.46
DC 8500	Bis (C13-15 Alkoxy) PG Amodimethicone	Dow corning	0.00	0.00	7.3
Dimethicone 9045	Cyclopentasiloxane and Dimethicone Crosspolymer	Dow corning	0.00	0.00	5.84
Water and minors			To 100	To 100	To 100

Example B phase separated after 24 hours storage at room temperature.

5 Example A and Example 1 remained stable after one weeks storage at 45°C

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Table 2

			Example C	Example 2
Trade name	INCI name	Supplier	Activity (%) in Formula	Activity (%) in Formula
Genamin BTLF	Behenytrimonium Chloride	Clariant	1.61	1.288
Lexamine s-13	Steramidopropyl dimethylamine	Inolex	0.38	0.3
Lanette S3	Cetearyl alcohol	Ecogreen Oleo chemicals	4.00	3.2
DC 5-7134 9:1 600K/8566 CTAC 70%	Dimethicone/amodimethicone/ Cetrimonium Chloride	Dow corning	3.50	2.8
Iron(II) D-gluconate dihydrate	Iron(II) D-gluconate dihydrate	Sigma-Aldrich	4.00	4.00
Aerosil R805			0.00	1.46
DC 8500			0.00	7.3
Dimethicone 9045			0.00	5.84
Water and minors			To 100	To 100

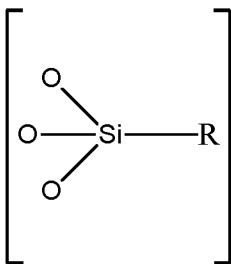
Example C phase separated after 24 hours storage at room temperature.

5 Example 2 remained stable.

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CLAIMS

1. A hair colouring composition comprising an emulsion comprising:
 - a) hydrophobically modified particles selected from the group consisting of silica, metal oxide and mixtures thereof;
 - b) amino functionalised silicone;
 - c) an iron salt or complex; and
 - d) water.
2. A composition according to any preceding claim further comprising a conditioning phase.
3. A composition according to claim 2 in which the conditioning phase comprises a cationic surfactant.
4. A composition according to any one of claims 2 or 3 comprising a fatty alcohol.
5. A composition according to any preceding claim in which the hydrophobically particles comprise silica.
6. A composition according to claim 5 in which the hydrophobically modified silica comprise

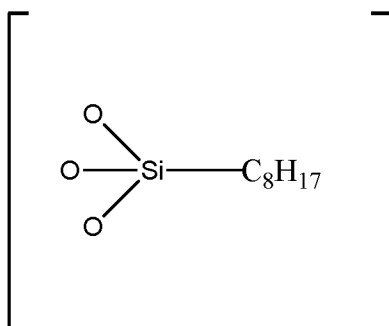


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in which R is a C₄ to C₁₈ alkyl group.

7. A composition according to claim 6 in which the hydrophobically modified silica comprises the group:

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8. A composition according to any preceding claim in which the hydrophobically modified particle has a primary particle size from 1nm to 100 nm.

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9. A composition according to any preceding claim in which the amino functionalized silicone comprises an amino glycol copolymer.

10. A composition according to any preceding claim in which the weight ratio of hydrophobically modified particle to amino functionalised silicone is from 3:1 to 1:50.

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11. A composition according to any one of the preceding claims wherein the emulsion further comprises at least one non amino functionalised silicone.

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12. A kit for colouring hair comprising:
- (i) a first composition comprising a polyphenol or source of polyphenol
 - (ii) a second composition as claimed in any preceding claim.

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13. A kit according to any claim in which the polyphenol is selected from the group consisting of gallic acid, methyl gallate, ethyl gallate , propyl gallate or mixtures thereof.
- 5 14. A method for colouring hair comprising the steps of applying to the hair a composition or kit described in any previous claim.
15. A method of colouring hair according to claim 14 comprising the steps of applying to hair in any order:
 - 10 i) a composition first composition comprising a polyphenol or source of polyphenol
 - ii) a second composition as described in any one of claims 1 to 11.