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(54) **Title:** USE OF COLOURED OR FLUORESCENT HYBRID PARTICLES FOR TREATING KERATIN FIBRES

(57) **Abstract:** The invention relates to the use, for dyeing keratin fibres, especially the hair, of hybrid particles obtained from an organosilane compound bearing a coloured species. The present invention also relates to a process for dyeing keratin fibres, especially the hair, which comprises the application to the fibres of a composition comprising, in a cosmetic medium, hybrid particles obtained from an organosilane compound bearing a coloured species. The particles that are useful for the present invention make it possible to obtain, depending on the coloured species bound to the organosilane, fast dyeing or optical lightening of keratin fibres.

USE OF COLOURED OR FLUORESCENT HYBRID PARTICLES FOR TREATING KERATIN FIBRES

The invention relates to the use of coloured or fluorescent organosilane particles in cosmetics, especially in the field of hair dyeing.

In the field of dyeing keratin fibres, in particular human keratin fibres such as the hair, two main modes of dyeing exist:

- *direct dyeing* or *semi-permanent dyeing* consists in providing colour via a coloured molecule that is adsorbed onto the surface of the keratin fibres and/or that penetrates by diffusion into the surface layers thereof. The leave-on times are generally fairly short and the mild dyeing conditions preserve the integrity of the keratin fibres, but the colorations obtained via this mode of dyeing show poor wash-fastness and fade out after only four or five shampoo washes. Furthermore, the ranges of shades obtained are generally limited;

- *oxidation dyeing* or *permanent dyeing* uses the oxidative condensation of colourless or weakly coloured molecules, known as oxidation bases, such as ortho- or para-phenylenediamines, ortho- or para-aminophenols or heterocyclic compounds in the presence of an oxidizing agent. This reaction leads to the formation of coloured polymer compounds in the keratin fibres. The main advantage of oxidation dyeing lies in the longevity of the colorations obtained, in particular in the resistance to washing and to external agents such as light, bad weather, permanent waving, perspiration and rubbing, and also in the production of a wide range of shades. However, the chemical dyeing conditions, such as the pH and an oxidizing medium, result in degradation of the keratin fibres. Moreover, this mode of dyeing requires relatively long leave-on times.

It is already known practice to use in the cosmetic and

medical fields sol-gel particles in which agents are encapsulated. In particular, document WO 2004/081 222 describes sol-gel particles in which dyes or fluorescent dyes are encapsulated, these particles possibly being used in haircare products or medical products.

The problem lies in the fact that since the particles are porous, it is found that, when these particles are used in cosmetic compositions, migration of the dyes outside the particles is observed, which firstly limits the gains in stability, especially with respect to light, that might be obtained, and secondly reduces the harmlessness of the system.

Hybrid sol-gel nanoparticles constituted of a colloidal silica, in which this silica is covalently bonded to fluorescent dyes, are moreover known. Such nanoparticles may also have a core/shell structure, the core being obtained from a standard alkoxysilane in sol-gel synthesis. Such particles are described, for example, in "Multicolour FRET silica nanoparticles by single wavelength excitation", Lin Wang and Weihong Tan, Nano Letters, 2006, vol. 6, No 1, 84-88; in "Bright and stable core-shell fluorescent silica nanoparticles", Nano Letters, 2005, vol. 5, No 1, 113-117. These nanoparticles are recommended for use in biological probes by virtue of their very strong fluorescent power.

The aim of the present invention is to provide a novel system for dyeing keratin fibres that does not have the drawbacks of the existing systems. In particular, the aim of the present invention is to provide a novel dye system that simultaneously has the advantages of harmlessness, stability and intensity of the colorations obtained, and gentleness on the keratin fibre, and which allows a wide range of shades to be obtained.

This aim is achieved with the present invention, one subject of which is the use for dyeing keratin fibres, especially the hair, of hybrid particles obtained from an organosilane compound bearing a coloured species.

A subject of the present invention is also a process for dyeing keratin fibres, especially the hair, which comprises the application to the fibres of a composition comprising, in a cosmetic medium, hybrid particles obtained from an organosilane compound bearing a coloured species.

The particles that are useful for the present invention make it possible to obtain, depending on the coloured species attached to the organosilane, fast dyeing or optical lightening of keratin fibres. Moreover, the present invention makes it possible to obtain fast dyeing or optical lightening without degradation of the keratin fibres.

For the purposes of the present invention, the term "optical lightening" means a visual lightening effect on naturally or artificially coloured keratin fibres, without using compounds that destroy the natural or artificial coloured pigments present in the keratin fibres.

For the purposes of the present invention, the expression "organosilane bearing a coloured species" means that the coloured species is connected via a covalent bond to the silicon of the organosilane.

The coloured species may be any radical derived from direct dyes, pigments or fluorescent dyes that are standard in the field of dyeing. Examples that may be mentioned include pigments, direct dyes and fluorescent dyes conventionally used in hair dyeing. Mention may be made especially of dyes of the nitrobenzene,

anthraquinone, nitropyridine, azo, cationic azo, xanthene, acridine, azine and nitrobenzene triarylmethane type, or natural dyes.

Among the benzene direct dyes that may be mentioned, in a non-limiting manner, are the following compounds: 1,4-diamino-2-nitrobenzene, 1-amino-2-nitro-4-β-hydroxyethylaminobenzene, 1-amino-2-nitro-4-bis(β-hydroxyethyl)-aminobenzene, 1,4-bis(β-hydroxyethyl-amino)-2-nitrobenzene, 1-β-hydroxyethylamino-2-nitro-4-bis(β-hydroxyethylamino)benzene, 1-β-hydroxyethylamino-2-nitro-4-aminobenzene, 1-β-hydroxyethylamino-2-nitro-4-(ethyl)(β-hydroxyethyl)aminobenzene, 1-amino-3-methyl-4-β-hydroxyethylamino-6-nitrobenzene, 1-amino-2-nitro-4-β-hydroxyethylamino-5-chlorobenzene, 1,2-diamino-4-nitrobenzene, 1-amino-2-β-hydroxyethylamino-5-nitrobenzene, 1,2-bis(β-hydroxyethylamino)-4-nitro-benzene, 1-amino-2-tris(hydroxymethyl)ethylamino-5-nitrobenzene, 1-hydroxy-2-amino-5-nitrobenzene, 1-hydroxy-2-amino-4-nitrobenzene, 1-hydroxy-3-nitro-4-aminobenzene, 1-hydroxy-2-amino-4,6-dinitrobenzene, 1-β-hydroxyethyloxy-2-β-hydroxyethylamino-5-nitrobenzene, 1-methoxy-2-β-hydroxyethylamino-5-nitrobenzene, 1-β-hydroxyethyloxy-3-methylamino-4-nitrobenzene, 1-β,γ-dihydroxypropyloxy-3-methylamino-4-nitrobenzene, 1-β-hydroxyethylamino-4-β,γ-dihydroxypropyloxy-2-nitro-benzene, 1-β,γ-dihydroxypropylamino-4-trifluoromethyl-2-nitrobenzene, 1-β-hydroxyethylamino-4-trifluoro-methyl-2-nitrobenzene, 1-β-hydroxyethylamino-3-methyl-2-nitrobenzene, 1-β-aminoethylamino-5-methoxy-2-nitro-benzene, 1-hydroxy-2-chloro-6-ethylamino-4-nitro-benzene, 1-hydroxy-2-chloro-6-amino-4-nitrobenzene, 1-hydroxy-6-bis(β-hydroxyethyl)amino-3-nitrobenzene, 1-β-hydroxyethylamino-2-nitrobenzene, 1-hydroxy-4-β-hydroxyethylamino-3-nitrobenzene.

Among the azo direct dyes that may be mentioned are the cationic azo dyes described in patent applications WO 95/15144, WO 95/01772 and EP-714 954, the content of

which forms an integral part of the invention.

Among these compounds, mention may be made most particularly of the following dyes: 1,3-dimethyl-2-[[4-(dimethylamino)phenyl]azo]-1H-imidazolium chloride, 1,3-dimethyl-2-[(4-aminophenyl)azo]-1H-imidazolium chloride, 1-methyl-4-[(methylphenylhydrazono)methyl]-pyridinium methyl sulfate.

Among the azo direct dyes, mention may also be made of the following dyes, described in the Colour Index International 3rd edition:
Disperse Red 17, Acid Yellow 9, Acid Black 1, Basic Red 22, Basic Red 76, Basic Yellow 57, Basic Brown 16, Acid Yellow 36, Acid Orange 7, Acid Red 33, Acid Red 35, Basic Brown 17, Acid Yellow 23, Acid Orange 24, Disperse Black 9.

Mention may also be made of 1-(4'-aminodiphenylazo)-2-methyl-4-bis(β-hydroxyethyl)aminobenzene and 4-hydroxy-3-(2-methoxyphenylazo)-1-naphthalenesulfonic acid.

Among the quinoline direct dyes that may be mentioned are the following dyes:
Disperse Red 15, Solvent Violet 13, Acid Violet 43, Disperse Violet 1, Disperse Violet 4, Disperse Blue 1, Disperse Violet 8, Disperse Blue 3, Disperse Red 11, Acid Blue 62, Disperse Blue 7, Basic Blue 22, Disperse Violet 15, Basic Blue 99, and also the following compounds:
1-N-methylmorpholiniumpropylamino-4-hydroxyanthraquinone, 1-aminopropylamino-4-methylaminoanthraquinone, 1-aminopropylaminoanthraquinone, 5-β-hydroxyethyl-1,4-diaminoanthraquinone, 2-aminoethylaminoanthraquinone, 1,4-bis(β,γ-dihydroxypropylamino)anthraquinone.

Among the azine dyes, mention may be made of the following compounds:
- Basic Blue 17 and Basic Red 2.

Among the triarylmethane dyes, mention may be made of the following compounds:
Basic Green 1, Acid Blue 9, Basic Violet 3, Basic Violet 14, Basic Blue 7, Acid Violet 49, Basic Blue 26, Acid Blue 7.

Among the indoamine dyes, mention may be made of the following compounds:
- 2-β-hydroxyethylamino-5-[bis(β-4'-hydroxyethyl)-amino]anilino-1,4-benzoquinone,
- 2-β-hydroxyethylamino-5-(2'-methoxy-4'-amino)anilino-1,4-benzoquinone,
- 3-N-(2'-chloro-4'-hydroxy)phenylacetylamino-6-methoxy-1,4-benzoquinone imine,
- 3-N-(3'-chloro-4'-methylamino)phenylureido-6-methyl-1,4-benzoquinone imine,
- 3-[4'-N-(ethyl, carbamylmethyl)amino]phenylureido-6-methyl-1,4-benzoquinone imine.

Among the natural direct dyes that may be used according to the invention, mention may be made of lawsone, juglone, alizarin, purpurin, carminic acid, kermesic acid, purpurogallin, protocatechaldehyde, indigo, isatin, curcumin, spinulosin and apigenidin.

Useful fluorescent dyes that may be mentioned include the fluorescent dyes mentioned in the "Molecular Probes Handbook of Fluorescent Probes and Research Chemical", 6th edition, R.P. Haugland, ed. (1996) or in Ullmann's Encyclopaedia of Industrial Chemistry, 2004 release, 7th edition, in the chapter "Fluorescent Dyes".

Conventionally, fluorescent dyes are aromatic or heteroaromatic compounds such as pyrenes, anthracenes, naphthalenes, acridines, indoles, benzindoles, ketopyrroles, oxazoles, benzoxazoles, diazoles, methines, carbocyanins, carbostyryls, porphyrins, salicylates, anthranilates, azulenes, perylenes, pyrazines, pyridines, quinolines, coumarins and

xanthenes.

As examples of functionalized dyes for obtaining an organosilane bearing a coloured species, mention may be made of tetramethylrhodamine 5- (and 6-)isothiocyanate (TRITC), Alexa Fluor® 488 C5 Maleimide, Alexa Fluor® 488 carboxylic acid, and succinimidyl ester.

The organosilanes may be represented by the general formula $R_{(4-n)}SiX_n$ in which X represents a hydrolysable group such as methoxy, ethoxy or 2-methoxyethoxy; R is a monovalent organic radical containing from 1 to 12 carbon atoms possibly containing functional organic groups capable of reacting with the coloured species, such as mercapto, epoxy, acrylyl, methacrylyl or amino; and n is an integer ranging from 0 to 4, and preferably n is equal to 3, with the condition that at least one organosilane used to form the particles comprises a group R containing a functional organic group capable of reacting with the coloured species. As examples of silanes capable of reacting to obtain a silane bearing a coloured species, mention may be made of 3-amino-propyltriethoxysilane and 3-mercaptopropyltriethoxy-silane.

According to one particular embodiment, the hybrid particles are hybrid particles whose coloured species is fluorescent.

According to one variant, the hybrid particles are covered with a silica sol-gel shell obtained from alkoxysilane, preferably trialkoxysilanes or tetraalkoxysilanes. The particles may be constituted, for example, of a homogeneous core and of one or more layers covering this core and forming the shell, these layers possibly being of different nature.

According to one particular embodiment of this variant, the hybrid particles are fluorescent particles

comprising a core constituted of organosilane(s) bearing fluorescent compound(s) and a silica shell. The core of the particles may then be obtained by reacting one or more fluorescent dyes with a reactive organosilane capable of reacting with this or these fluorescent dye(s). The core is then obtained via a condensation reaction of one or more (organo)alkoxysilanes. These (organo)alkoxysilanes may make it possible to obtain one or more layers constituting the shell, these layers possibly having particular different properties as a function of the (organo)alkoxysilane used, the thickness of the shell, the continuity of the shell on the core, and the ratio of the thickness of the shell to the core.

The size of the particles that are useful in the present invention may vary widely. However, these particles are generally nanoparticles, especially nanoparticles with a number-average diameter ranging from 1 to 100 nm and preferably between 1 and 50 nm. In a particularly preferred manner, these particles are nanoparticles with a diameter of between 1 and 20 nm.

Preferably, these particles are monodisperse.

In the context of the invention, the size and the size distribution are determined by electron microscopy (SEM).

The hybrid particles that are useful in the present invention are known particles of the art obtained via a sol/gel route. For example, these particles are described in the Nano Letters articles mentioned previously and also in patent application US 2004/0 101 822. For further details regarding the nature of the precursors and the reaction mechanisms in sol-gel processes, reference may be made to the book “Sol Gel Science” published by C.J. Brinker and G.W. Scherer, Academic Press publications (ISBN 0-12-134970-

5).

The hybrid particles are generally obtained via a sol-gel process that is performed using (organo)silanes. These organosilanes may be represented by the general formula $R_{(4-n)}SiX_n$ as defined above.

Thus, it is possible to obtain fluorescent hybrid particles that are useful in the present invention by reacting and condensing the reactive fluorescent dye tetramethylrhodamine isothiocyanate (TRITC) with a reactive organosilane such as 3-aminopropyl-triethoxysilane to form hybrid particles rich in fluorescent dye, the fluorescent dye being covalently bonded to the silica derivative thus formed. One or two steps may be performed depending on whether or not the organosilane bearing a coloured species is isolated.

To obtain the particles with a core and shell as described above, this first particle that will become the core is reacted with an alkoxysilane, preferably a tetraalkoxysilane such as tetraethyl orthosilicate, which, via a condensation reaction, will form a dense silica shell around the fluorescent core.

By way of example, to obtain hybrid particles, TRITC and 3-aminopropyltriethoxysilane are reacted via an addition reaction in a mole ratio of 1:50 in a moisture-free medium. The product of this reaction is then placed in a reactor in the presence of aqueous ammonia, water and solvent. The reaction is left to proceed overnight. After this reaction, tetraethyl orthosilicate is introduced into the reaction medium in order to form the silica shell.

According to one variant, homogeneous particles may be obtained by simultaneously reacting via co-condensation all the reagents described above.

The particles may also be covered with one or more additional organic and/or mineral layers, preferably having affinity for the hair. Examples of organic layers that may be mentioned include the layers obtained with polyethylene glycol, polyurethane, dextrin, polyacrylic, polyvinylpyrrolidone, polyvinyl-caprolactone or a mixture of these materials.

Examples of mineral layers that may be mentioned include the layers obtained with alumina, silica or clay, or a mixture of these materials. These layers may be obtained via the sol-gel process using organosilane.

The particles that are useful in the present invention may be used incorporated in compositions. In this case, the composition contains a cosmetic medium that is suitable for dyeing, also known as a dye support, and generally consisting of water or of a mixture of water and of at least one organic solvent. Examples of organic solvents that may be mentioned include C_1-C_4 lower alkanols, such as ethanol and isopropanol; polyols and polyol ethers, for instance 2-butoxyethanol, propylene glycol, propylene glycol monomethyl ether, diethylene glycol monomethyl ether and monoethyl ether, and also aromatic alcohols, for instance benzyl alcohol or phenoxyethanol, and mixtures thereof.

The concentration of hybrid particles in the compositions used according to the invention preferably ranges from 0.001% to 99%, even more preferentially from 0.005% to 50% and ideally from 0.01% to 20%.

The solvents are preferably present in proportions preferably of between 1% and 40% by weight approximately, and even more preferentially between 5% and 30% by weight approximately, relative to the total weight of the dye composition.

The dye composition in accordance with the invention may also contain various adjuvants conventionally used in hair dye compositions, such as anionic, cationic, nonionic, amphoteric or zwitterionic surfactants or mixtures thereof, anionic, cationic, nonionic, amphoteric or zwitterionic polymers or mixtures thereof, mineral or organic thickeners, and in particular anionic, cationic, nonionic and amphoteric polymeric associative thickeners, antioxidants, penetrants, sequestrants, fragrances, buffers, dispersants, conditioning agents, for instance volatile or non-volatile, modified or unmodified silicones, film-forming agents, ceramides, preserving agents and opacifiers.

The above adjuvants are generally present in an amount for each of them of between 0.01% and 20% by weight relative to the weight of the composition.

The composition according to the present invention preferably comprises, in addition to the nanoparticles, a surfactant or a thickening polymer.

These surfactants and polymers may be of nonionic, anionic, cationic or amphoteric nature.

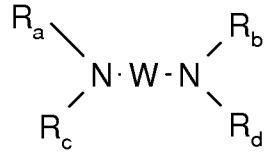
The amounts of surfactants and/or of thickening polymer present in the composition according to the invention may range from 0.01% to 40% and preferably from 0.5% to 30% of the total weight of the composition.

Needless to say, a person skilled in the art will take care to select this or these optional additional compound(s) such that the advantageous properties intrinsically associated with the oxidation dye composition in accordance with the invention are not, or are not substantially, adversely affected by the envisaged addition(s).

The pH of the dye composition in accordance with the invention is generally between 3 and 12 approximately and preferably between 5 and 11 approximately. It may be adjusted to the desired value by means of acidifying or basifying agents usually used in the dyeing of keratin fibres, or alternatively using standard buffer systems.

Among the acidifying agents, examples that may be mentioned include mineral or organic acids, for instance hydrochloric acid, orthophosphoric acid, sulfuric acid, carboxylic acids, for instance acetic acid, tartaric acid, citric acid or lactic acid, and sulfonic acids.

Among the basifying agents, examples that may be mentioned include aqueous ammonia, alkali metal carbonates, alkanolamines such as monoethanolamine, diethanolamine, triethanolamine and derivatives thereof, sodium hydroxide, potassium hydroxide and the compounds of formula (II) below:



(II)

in which W is a propylene residue optionally substituted with a hydroxyl group or a C_1-C_4 alkyl radical; R_a, R_b, R_c and R_d, which may be identical or different, represent a hydrogen atom or a C_1-C_4 alkyl or C_1-C_4 hydroxyalkyl radical.

The dye composition according to the invention may be in various forms, such as in the form of liquids, creams or gels, or in any other form that is suitable for dyeing keratin fibres, and especially human hair. It may be in the form of a dye, but also in the form of a shampoo, a hair conditioner, a lacquer or a hair-shaping composition.

Another subject of the invention is a process for treating keratin fibres, and in particular human keratin fibres such as the hair, which comprises the application to the fibres of a composition according to the present invention.

According to one particular embodiment, the process of the invention is a process for dyeing keratin fibres. The composition of the invention may be applied to wet or dry fibres. The application may be followed by a step of washing and/or rinsing the keratin fibres.

When the coloured species is a fluorescent dye, then the process of the invention, when it is applied to naturally or artificially coloured dark hair, for example hair with a tone depth of greater than or equal to 4 and preferably greater than or equal to 6, makes it possible to obtain optical lightening of the hair. Thus, without bleaching the keratin fibres, significant lightening of the fibres may be seen. The notion of "tone" is based on the classification of natural shades, one tone separating each shade from the one immediately following or preceding it. This definition and the classification of natural shades are well known to hairstyling professionals and are published in the book "Sciences des traitements capillaires [Hair treatment sciences]" by Charles Zviak, 1988, published by Masson, pp. 215 and 278.

As an example of coloration of keratin fibres, an aqueous composition containing 5% of alkylpolyglucoside and 0.5% of fluorescent red core/shell nanoparticles (TRITC), prepared according to the procedure described in Examples 1 and 3 (pp. 12-13) of patent US 2004/101 822, may be applied at room temperature to a lock of natural grey hair containing 90% white hairs. After the lock of hair treated in this way has been drained and dried, a red coloration of the lock of hair can be seen.

CLAIMS

1. Use, for dyeing keratin fibres, of hybrid particles obtained from an organosilane compound bearing a coloured species.

2. Use according to Claim 1, in which the coloured species is chosen from direct dyes of nitrobenzene, anthraquinone, nitropyridine, azo, cationic azo, xanthene, acridine, azine or nitrobenzene triarylmethane type and natural dyes.

3. Use according to Claim 1, in which the coloured species is a pigment.

4. Use according to Claim 1 or 2, in which the coloured species is a fluorescent dye.

5. Use according to Claim 4, in which the coloured species is a fluorescent dye chosen from pyrenes, anthracenes, naphthalenes, acridines, indoles, benzindoles, ketopyrroles, oxazoles, benzoxazoles, diazoles, methines, carbocyanins, carbostyryls, porphyrins, salicylates, anthranilates, azulenes, perylenes, pyrazines, pyridines, quinolines, coumarins and xanthenes.

6. Use according to any one of the preceding claims, in which the hybrid particles are obtained from an organosilane of formula $R_{(4-n)}SiX_n$ in which X represents a hydrolysable group; R is a monovalent organic radical containing from 1 to 12 carbon atoms, possibly containing functional organic groups capable of reacting with the coloured species; and n is an integer ranging from 0 to 4, on condition that at least one organosilane used to form the particles comprises a group R containing a functional organic group capable of reacting with the coloured species.

7. Use according to any one of the preceding claims, in which the hybrid particles are covered with a sol-gel silica shell obtained from alkoxysilane.

8. Use according to Claim 7, in which the shell is obtained from trialkoxysilanes and/or tetraalkoxysilanes.

9. Use according to either of Claims 7 and 8, in which the shell comprises one or more layers.

10. Use according to any one of Claims 1 to 9, in which the size of the nanoparticles is between 1 and 100 nm and preferably between 1 and 50 nm.

11. Use according to any one of the preceding claims, for dyeing the hair.

12. Dye composition comprising, in a suitable dyeing medium, at least one hybrid particle as defined in any one of the preceding claims.

13. Composition according to Claim 12, which comprises at least one surfactant and/or one thickening polymer.

14. Composition according to Claim 12 or 13, in the form of a dye, a shampoo, a hair conditioner, a lacquer or a hair-shaping composition.

15. Process for dyeing keratin fibres, which comprises the application to these fibres of a composition as defined in Claims 12 to 14.

16. Process for lightening keratin fibres, which comprises the application of a composition as defined in Claims 12 to 14 to keratin fibres with a tone depth of greater than or equal to 4 and preferably greater than or equal to 6.