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#### (54)**Fabric softening compositions**

(57)The present invention relates to a fabric softening composition comprising a fabric softener having at least two long chains and a cellulose reactive dye fixing agent, said composition providing effective dye fixing properties and softening properties to the treated fabric.

Also provided herein is a method for the domestic treatment of a fabric to reduce the amount of dye released from the fabric during wet treatments and comprising the step of contacting the fabrics with a cellulose reactive dye fixing agent or a fabric softening composition as defined herein; and thereafter subjecting the treated fabrics with a heating source in a dry medium.

### Description

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### Field of the invention

[0001] The present invention relates to a fabric softening composition and more particularly to a method and a composition which reduce the amount of dyes released from coloured fabrics upon subsequent wet treatments such as those which occur in a laundry operation.

### Background of the invention

**[0002]** The domestic treatment of colored fabric is a problem known in the art to the formulator of laundry compositions. More particularly, the problem of formulating laundry compositions which reduces the amount of dyes released from coloured fabrics upon wet treatment is a particular challenge to the formulator. This problem is now even more acute with the trends of consumers to move towards more colored fabrics.

[0003] Numerous solutions have been proposed in the art to solve this problem such as by treating the fabric with a dye scavenger during the washing process as exemplified by EP 0,341,205, EP 0,033,815 or with a polyvinyl substance as exemplified by WO 94/11482. However, all these solutions are focused on preventing the end result of the dye bleeding, that is the redeposition of the dye on the fabrics. It is now an object of the invention to take the problem of dye at its source, that is to prevent the dye from bleeding.

[0004] It is another object of the invention to formulate compositions which provide effective softening benefits on the treated fabrics.

**[0005]** One solution to these problems is by the preparation of specific dye fixing agents such as REWIN SRF having dye fixing properties as well as softness properties as described in Textilveredlung, Vol 25, no.2., 1990, pp 12.

[0006] Still another solution to these problems would be to use a cross-linker such Arkofix NZF commercially available from Hoechst. Cross-linkers are known in the field of dyes to provide an effective protection of the dyed fabrics by their property of being cellulose reactive. However, these compounds do not have a dye fixing property.

**[0007]** In addition, solutions may be found for use in the industrial treatments. However, these solutions are not usually transposable to domestic treatments. Indeed, in industrial processes a strict control over parameters such as pH, electrolyte concentration, water hardness, temperature, etc. is possible whereas in a domestic washing machine, such a high level of control is not possible.

**[0008]** In addition, in a domestic process, and in particular in a domestic rinse process, it is not practical to rely on high treatment temperatures such as those used in industrial processes, that is of above 40°C. Furthermore, industrial processes use high concentrations of fixing agents which is required for industrial scale treatment whilst for domestic treatment a low level is most preferred for economic reasons.

**[0009]** Accordingly, notwithstanding the advances in the art, there is still a need for a composition which provides effective and durable reduction of the amount of dyes released from coloured fabrics upon subsequent wet domestic treatments whilst still providing effective fabric softness.

**[0010]** EP 462806 provides the use of a cationic dye fixing agent in domestic treatment which assist in binding the loosely held dye to the fabric. However, the dye fixing agents described in this patent provide the fixation of the dye via electrostatic interactions with the dye and the cellulose fibre. These interactions are by definition reversible, and therefore labile.

**[0011]** The Applicant has now found that the use of a composition comprising a fabric softener having di-long chains, and a cellulose reactive dye fixing agent fulfills such a need. The reactive dye fixing agents used in the present invention react covalently with cellulose fibers, i.e. form a chemical bond that is more irreversible than mere ionic interactions, thereby providing more longevity to the dye fixative treatment.

**[0012]** It has also been advantageously found that the cellulose reactive dye fixing agents for use in the present invention even provide better performance than the dye fixing agents of EP 0,462,806.

**[0013]** By a cellulose reactive dye fixing agent, it is meant that a reactive functionality, which is grafted on the fixing agent, can form covalent bonds with cellulose. The reactivity can then further be improved upon heat treatment. A test method to define which compound are cellulose reactive dye fixing agent is given hereinafter.

**[0014]** Accordingly, it is therefore an advantage of the invention to provide compositions with effective and durable dye fixing properties together with softening properties.

**[0015]** Another advantage of the invention is that such compositions provide an increase performance of the above mentioned benefit after subjecting the treated fabrics with a heating source.

[0016] A further advantage of the invention is that the treated fabrics will thereafter show a reduced tendency in the subsequent wash to release dye. Such benefit is more particularly seen after multi-wash cycles.

### Summary of the invention

[0017] The present invention relates to a fabric softening composition comprising a fabric softener having at least two long chains and a cellulose reactive dye fixing agent.

[0018] In another aspect of the invention is provided a method for the domestic treatment of a fabric to reduce the amount of dye released from the fabric during wet treatments and comprising the step of contacting the fabrics with a cellulose reactive dye fixing agent or composition containing said agent; and thereafter contacting the treated fabrics with a heating source in a dry medium.

#### 10 Detailed description of the invention

**[0019]** According to one aspect of the invention, there is provided a fabric softening composition with effective and durable dye fixing properties together with softening properties.

#### 15 Fabric softeners

[0020] An essential component of the composition according to the invention is a fabric softener component having at least two long chains. By component having at least two long chains is meant a component containing at least two alkyl or alkenyl chains, each comprising from 10 to 25 carbon atoms. Such fabric softener provides effective softness benefit to the treated fabrics.

**[0021]** Typical levels of said fabric softener components within the liquid softener compositions are from 1% to 80% by weight of the compositions. Depending on the composition execution which can be dilute with a preferred level of fabric softening components from 1% to 5%, or concentrated, with a preferred level of fabric softening components from 5% to 80%, more preferably 10% to 50%, most preferably 15% to 35% by weight.

Where the fabric softener composition is applied on a substrate such as a dryer-sheet, the preferred level of fabric softener components will preferably be from 20% to 99%, more preferably from 30% to 90% by weight, and even more preferably from 35% to 80% by weight.

**[0022]** Typical cationic fabric softening components having di-long chains include the water-insoluble quaternary-ammonium fabric softening actives, the most commonly used having been di-long alkyl chain ammonium chloride. Preferred cationic softeners among these include the following:

- 1) ditallow dimethylammonium chloride (DTDMAC);
- 2) dihydrogenated tallow dimethylammonium chloride;
- 3) dihydrogenated tallow dimethylammonium methylsulfate;
- distearyl dimethylammonium chloride;
- 5) dioleyl dimethylammonium chloride;
- 6) dipalmityl hydroxyethyl methylammonium chloride;
- 7) stearyl benzyl dimethylammonium chloride;
- 8) tallow trimethylammonium chloride;
- hydrogenated tallow trimethylammonium chloride;
  - 10) C<sub>12-14</sub> alkyl hydroxyethyl dimethylammonium chloride;
  - 11) C<sub>12-18</sub> alkyl dihydroxyethyl methylammonium chloride;
  - 12) di(stearoyloxyethyl) dimethylammonium chloride (DSOEDMAC);
  - 13) di(tallowoyloxyethyl) dimethylammonium chloride;
- 14) ditallow imidazolinium methylsulfate;
  - 15) 1-(2-tallowylamidoethyl)-2-tallowyl imidazolinium methylsulfate.

**[0023]** However, in recent years, the need has arisen for more environmentally-friendly materials, and rapidly biodegradable quaternary ammonium compounds have been presented as alternatives to the traditionally used di-long chain ammonium chlorides. Such quaternary ammonium compounds contain long chain alk(en)yl groups interrupted by functional groups such as carboxy groups. Said materials and fabric softening compositions containing them are disclosed in numerous publications such as EP-A-0,040,562, and EP-A-0,239,910.

[0024] The preferred fabric softening agents to be used in the present invention compositions are quaternary ammonium compounds or amine precursors herein having the formula (II) or (III), below:

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Q is -O-C(O)- or -C(O)-O- or -O-C(O)-O- or -NR<sup>4</sup>-C(O)- or -C(O)-NR<sup>4</sup>-;  $\begin{array}{c} R^1 \text{ is } (CH_2)_n\text{-}Q\text{-}T^2 \text{ or } T^3 \text{ or } R^3; \\ R^2 \text{ is } (CH_2)_m\text{-}Q\text{-}T^4 \text{ or } T^5 \text{ or } R^3; \\ R^3 \text{ is } C_1\text{-}C_4 \text{ alkyl or } C_1\text{-}C_4 \text{ hydroxyalkyl or } H; \\ 20 & R^4 \text{ is } H \text{ or } C_1\text{-}C_4 \text{ alkyl or } C_1\text{-}C_4 \text{ hydroxyalkyl;} \\ T^1, T^2, T^3, T^4, T^5 \text{ are (the same or different) } C_{11}\text{-}C_{22} \text{ alkyl or alkenyl;} \\ n \text{ and } m \text{ are integers from 1 to 4; and} \\ X^- \text{ is a softener-compatible anion, such as chloride, methyl sulfate, etc.} \end{array}$ 

The alkyl, or alkenyl, chain T<sup>1</sup>, T<sup>2</sup>, T<sup>3</sup>, T<sup>4</sup>, T<sup>5</sup> must contain at least 11 carbon atoms, preferably at least 16 carbon atoms. The chain may be straight or branched.

Q, n, T<sup>1</sup>, and T<sup>2</sup> may be the same or different when more than one is present in the molecule.

Tallow is a convenient and inexpensive source of long chain alkyl and alkenyl material. The compounds wherein  $T^1$ ,  $T^2$ ,  $T^3$ ,  $T^4$ ,  $T^5$  represents the mixture of long chain materials typical for tallow are particularly preferred.

Specific examples of quaternary ammonium compounds suitable for use in the aqueous fabric softening compositions herein include:

N,N-di(tallowyl-oxy-ethyl)-N,N-dimethyl ammonium chloride;

N,N-di(canolyl-oxy-ethyl)-N,N-dimethyl ammonium chloride;

N,N-di(tallowyl-oxy-ethyl)-N-methyl, N-(2-hydroxyethyl) ammonium chloride;

N,N-di(canolyl-oxy-ethyl)-N-methyl, N-(2-hydroxyethyl) ammonium chloride;

N,N-di(2-tallowyloxy-2-oxo-ethyl)-N,N-dimethyl ammonium chloride;

N,N-di(2-canolyloxy-2-oxo-ethyl)-N,N-dimethyl ammonium chloride

N,N-di(2-tallowyloxyethylcarbonyloxyethyl)-N,N-dimethyl ammonium chloride;

40 N,N-di(2-canolyloxyethylcarbonyloxyethyl)-N,N-dimethyl ammonium chloride;

N-(2-tallowoyloxy-2-ethyl)-N-(2-tallowyloxy-2-oxo-ethyl)-N,N-dimethyl ammonium chloride;

N-(2-canolyloxy-2-ethyl)-N-(2-canolyloxy-2-oxo-ethyl)-N,N-dimethyl ammonium chloride;

N,N,N-tri(tallowyl-oxy-ethyl)-N-methyl ammonium chloride;

N,N,N-tricanolyl-oxy-ethyl)-N-methyl ammonium chloride;

N-(2-tallowyloxy-2-oxoethyl)-N-(tallowyl)-N,N-dimethyl ammonium chloride;

N-(2-canolyloxy-2-oxoethyl)-N-(canolyl)-N,N-dimethyl ammonium chloride;

1,2-ditallowyloxy-3-N,N,N-trimethylammoniopropane chloride; and

1,2-dicanolyloxy-3-N,N,N-trimethylammoniopropane chloride;

and mixtures of the above actives.

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**[0025]** Particularly preferred is N,N-di(tallowoyl-oxy-ethyl)-N,N-dimethyl ammonium chloride, where the tallow chains are at least partially unsaturated.

The level of unsaturation of the tallow chain can be measured by the lodine Value (IV) of the corresponding fatty acid, which in the present case should preferably be in the range of from 5 to 100 with two categories of compounds being distinguished, having a IV below or above 25.

Indeed, for compounds of Formula (II) made from tallow fatty acids having a IV of from 5 to 25, preferably 15 to 20, it has been found that a cis/trans isomer weight ratio greater than about 30/70, preferably greater than about 50/50 and more preferably greater than about 70/30 provides optimal concentrability.

For compounds of Formula (II) made from tallow fatty acids having a IV of above 25, the ratio of cis to trans isomers has been found to be less critical unless very high concentrations are needed.

Other examples of suitable quaternary ammoniums of Formula (II) and (III) are obtained by, e.g.,

- replacing "tallow" in the above compounds with, for example, coco, palm, lauryl, oleyl, ricinoleyl, stearyl, palmityl,
   or the like, said fatty acyl chains being either fully saturated, or preferably at least partly unsaturated;
  - replacing "methyl" in the above compounds with ethyl, ethoxy, propyl, propoxy, isopropyl, butyl, isobutyl or t-butyl;
  - replacing "chloride" in the above compounds with bromide, methylsulfate, formate, sulfate, nitrate, and the like.
- [0026] In fact, the anion is merely present as a counterion of the positively charged quaternary ammonium compounds. The nature of the counterion is not critical at all to the practice of the present invention. The scope of this invention is not considered limited to any particular anion.
  - By "amine precursors thereof' is meant the secondary or tertiary amines corresponding to the above quaternary ammonium compounds, said amines being substantially protonated in the present compositions due to the claimed pH values.
  - The quaternary ammonium or amine precursors compounds herein are present at levels of from about 1% to about 80% of compositions herein, depending on the composition execution which can be dilute with a preferred level of active from about 5% to about 15%, or concentrated, with a preferred level of active from about 15% to about 50%, most preferably about 15% to about 35%.
- For the preceding fabric softening agents, the pH of the compositions herein is an important parameter of the present invention. Indeed, it influences the stability of the quaternary ammonium or amine precursors compounds, especially in prolonged storage conditions.
  - The pH, as defined in the present context, is measured in the neat compositions at 20 °C. While these compositions are operable at pH of less than about 6.0, for optimum hydrolytic stability of these compositions, the neat pH, measured in the above-mentioned conditions, must be in the range of from about 2.0 to about 4.5, preferably about 2.0 to about 3.5. The pH of these compositions herein can be regulated by the addition of a Bronsted acid.
  - Examples of suitable acids include the inorganic mineral acids, carboxylic acids, in particular the low molecular weight  $(C_1-C_5)$  carboxylic acids, and alkylsulfonic acids. Suitable inorganic acids include HCl,  $H_2SO_4$ ,  $HNO_3$  and  $H_3PO_4$ . Suitable organic acids include formic, acetic, citric, methylsulfonic and ethylsulfonic acid. Preferred acids are citric, hydrochloric, phosphoric, formic, methylsulfonic acid, and benzoic acids.

### Cellulose reactive dye fixing agents

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[0027] The other essential component of the invention is a cellulose reactive dye fixing agent. Typical levels of such agents are from 0.01% to 50%, preferably from 0.01% to 25%, more preferably from 0.05 to 10% by weight, most preferably from 0.5 to 5% by weight of the composition.

**[0028]** By "cellulose reactive dye fixing agent", it is meant that the agent reacts with the cellulose fibers upon heat treatment. The agents suitable for use herein can be defined by the following test procedure, so called cellulose reactivity test measurement.

### Cellulose reactivity test measurement

[0029] Two pieces of bleeding fabrics (e.g. 10 x 10 cm of knitted cotton dyed with Direct Red 80) are soaked for 20 minutes in an aqueous solution of 1% (w/w) of the cellulose reactive dye fixing agent candidate. The pH of the solution is as it is obtained at this concentration.

[0030] The swatches are then dried. One of the dried swatches as well as an unsoaked swatch (control 1) are passed 10 times through an ironing calender set on a linen setting.

[0031] A control 2 swatch is also used in this measurement test which is a non-soaked and non-ironed swatch.

[0032] The 4 swatches are washed separately in Launder-o-meter pots under typical conditions with a commercial detergent used at the recommended dosage for ½ hour at 60°C, followed by a thorough rinsing of 4 times 200 ml of cold water and then line dried.

[0033] The wash-fastness is then measured on the swatches by determination of their so-called delta-E values versus a new, untreated swatch. Delta E's are defined, for instance, in ASTM D2244. Delta E is the computed color difference as defined in ASTM D2244, i.e the magnitude and direction of the difference between two psychophysical color stimuli defined by tristimulus values, or by chromaticity coordinates and luminance factor, as computed by means of a specified set of color-difference equations defined in the CIE 1976 CIELAB opponent-color space, the Hunter opponent-color space, the Friele-Mac Adam-Chickering color space or any equivalent color space.

[0034] Accordingly, the lower the Delta E versus new, the better the wash fastness improvement.

[0035] If the washfastness improvement of the ironed-soaked swatch is better than that of the non-ironed soak swatch and also better than the two respective control 1 and 2, then the candidate is a cellulose reactive dye fixing agent for the purpose of the invention.

**[0036]** Typical cellulose reactive dye fixing agents are products containing the reactive group of the reactive dye classes selected from halogeno-triazine products, vinyl sulphones compounds, epichlorhydrine derivatives, hydroxyethylene urea derivatives, formaldehyde condensation products, polycarboxylates, glyoxal and glutaraldehyde derivatives and mixtures thereof.

**[0037]** Other reactive functionalities for cellulose can be found in Textile processing and properties. Elsevier (1997) from Tyrone L. Vigo at page 120 to 121, which provides the use of specific electrophilic groups with cellulose affinity.

[0038] Preferred hydroxyethylene urea derivatives include dimethyloldihydroxyethylene, urea, and dimethyl urea gly-

**[0039]** Preferred formaldehyde condensation products include the condensation products derived from formaldehyde and a group selected from an amino-group, an imino-group, a phenol group, an urea group, a cyanamide group and an aromatic group. Commercially available compounds among this class are Sandofix WE 56 from Clariant, Zetex E from Zeneca and Levogen BF from Bayer.

**[0040]** Preferred polycarboxylates derivatives include butane tetracarboxilic acid derivatives, citric acid derivatives, polyacrylates and derivatives thereof.

**[0041]** A most preferred cellulosic reactive dye fixing agents is one of the hydroxyethylene urea derivatives class commercialised under the tradename of Indosol CR from Clariant. Still other most preferred cellulosic reactive dye fixing agents are commercialised under the tradename Rewin DWR and Rewin WBS from CHT R. Beitlich.

**[0042]** The compositions of the invention also comprise optional ingredients, such as additional fabric softener materials, liquid carrier, concentration aids, other ingredients and mixtures thereof.

### 1. Additional fabric softener materials

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**[0043]** Additional fabric softening materials may be used in addition to the cationic fabric softener. These may be selected from nonionic, amphoteric or anionic fabric softening materials. Disclosure of such materials may be found in US 4,327,133; US 4,421,792; US 4,426,299; US 4,460,485; US 3,644,203; US 4,661,269; U.S 4,439,335; U.S 3,861,870; US 4,308,151; US 3,886,075; US 4,233,164; US 4,401,578; US 3,974,076; US 4,237,016 and EP 472,178.

[0044] Typically, such nonionic fabric softener materials have a HLB of from about 2 to about 9, more typically from about 3 to about 7. Such nonionic fabric softener materials tend to be readily dispersed either by themselves, or when combined with other materials such as single-long-chain alkyl cationic surfactant described in detail hereinafter. Dispersibility can be improved by using more single-long-chain alkyl cationic surfactant, mixture with other materials as set forth hereinafter, use of hotter water, and/or more agitation. In general, the materials selected should be relatively crystalline, higher melting, (e.g. >40°C) and relatively water-insoluble.

Preferred nonionic softeners are fatty acid partial esters of polyhydric alcohols, or anhydrides thereof, wherein the alcohol, or anhydride, contains from 2 to 18, preferably from 2 to 8, carbon atoms, and each fatty acid moiety contains from 12 to 30, preferably from 16 to 20, carbon atoms. Typically, such softeners contain from one to 3, preferably 2 fatty acid groups per molecule.

The polyhydric alcohol portion of the ester can be ethylene glycol, glycerol, poly (e.g., di-, tri-, tetra, penta-, and/or hexa-) glycerol, xylitol, sucrose, erythritol, pentaerythritol, sorbitol or sorbitan. Sorbitan esters and polyglycerol monostearate are particularly preferred.

The fatty acid portion of the ester is normally derived from fatty acids having from 12 to 30, preferably from 16 to 20, carbon atoms, typical examples of said fatty acids being lauric acid, myristic acid, palmitic acid, stearic acid and behenic acid.

Highly preferred optional nonionic softening agents for use in the present invention are the sorbitan esters, which are esterified dehydration products of sorbitol, and the glycerol esters.

Commercial sorbitan monostearate is a suitable material. Mixtures of sorbitan stearate and sorbitan palmitate having stearate/palmitate weight ratios varying between about 10:1 and about 1:10, and 1,5-sorbitan esters are also useful.

[0045] Glycerol and polyglycerol esters, especially glycerol, diglycerol, triglycerol, and polyglycerol mono- and/or diesters, preferably mono-, are preferred herein (e.g. polyglycerol monostearate with a trade name of Radiasurf 7248). Useful glycerol and polyglycerol esters include mono-esters with stearic, oleic, palmitic, lauric, isostearic, myristic, and/or behenic acids and the diesters of stearic, oleic, palmitic, lauric, isostearic, behenic, and/or myristic acids. It is understood that the typical mono-ester contains some di- and tri-ester, etc.

The "glycerol esters" also include the polyglycerol, e.g., diglycerol through octaglycerol esters. The polyglycerol polyols are formed by condensing glycerin or epichlorohydrin together to link the glycerol moieties via ether linkages. The mono- and/or diesters of the polyglycerol polyols are preferred, the fatty acyl groups typically being those described hereinbefore for the sorbitan and glycerol esters.

### 2. Liquid carrier

[0046] Another optional, but preferred, ingredient is a liquid carrier. The liquid carrier employed in the instant compositions is preferably at least primarily water due to its low cost, relative availability, safety, and environmental compatibility. The level of water in the liquid carrier is preferably at least about 50%, most preferably at least about 60%, by weight of the carrier. Mixtures of water and low molecular weight, e.g., <about 200, organic solvent, e.g., lower alcohols such as ethanol, propanol, isopropanol or butanol are useful as the carrier liquid. Low molecular weight alcohols include monohydric, dihydric (glycol, etc.) trihydric (glycerol, etc.), and higher polyhydric (polyols) alcohols.

#### 10 3. Concentration aids

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**[0047]** Concentrated compositions of the present invention may require organic and/or inorganic concentration aids to go to even higher concentrations and/or to meet higher stability standards depending on the other ingredients. Surfactant concentration aids are typically selected from the group consisting of single long chain alkyl cationic surfactants; nonionic surfactants; amine oxides; fatty acids; or mixtures thereof, typically used at a level of from 0 to 15% of the composition.

### Single long chain alkyl cationic surfactants

20 **[0048]** Such mono-long-chain-alkyl cationic surfactants useful in the present invention are, preferably, quaternary ammonium salts of the general formula:

$$[R^2N^+R^3]X^-$$

wherein the R<sup>2</sup> group is C<sub>10</sub>-C<sub>22</sub> hydrocarbon group, preferably C<sub>12</sub>-C<sub>18</sub> alkyl group of the corresponding ester linkage interrupted group with a short alkylene (C<sub>1</sub>-C<sub>4</sub>) group between the ester linkage and the N, and having a similar hydrocarbon group, e.g., a fatty acid ester of choline, preferably C<sub>12</sub>-C<sub>14</sub> (coco) choline ester and/or C<sub>16</sub>-C<sub>18</sub> tallow choline ester at from 0.1% to 20% by weight of the softener active. Each R<sup>3</sup> is a C<sub>1</sub>-C<sub>4</sub> alkyl or substituted (e.g., hydroxy) alkyl, or hydrogen, preferably methyl, and the counterion X<sup>-</sup> is a softener compatible anion, for example, chloride, bromide, methyl sulfate, etc.

**[0049]** Other cationic materials with ring structures such as alkyl imidazoline, imidazolinium, pyridine, and pyridinium salts having a single  $C_{12}$ - $C_{30}$  alkyl chain can also be used. Very low pH is required to stabilize, e.g., imidazoline ring structures.

Some alkyl imidazolinium salts and their imidazoline precursors useful in the present invention have the general formula :

$$CH_2 - CH_2$$
 $N + C_2H_4 - Y^2 - R^7$ 
 $X^{-1}$ 
 $R^{8}$ 

wherein Y<sup>2</sup> is -C(O)-O-, -O-(O)C-, -C(O)-N(R<sup>5</sup>)-, or -N(R<sup>5</sup>)-C(O)- in which R<sup>5</sup> is hydrogen or a C<sub>1</sub>-C<sub>4</sub> alkyl radical; R<sup>6</sup> is a C<sub>1</sub>-C<sub>4</sub> alkyl radical or H (for imidazoline precursors); R<sup>7</sup> and R<sup>8</sup> are each independently selected from R<sup>3</sup> and R<sup>2</sup> as defined hereinbefore for the single-long-chain cationic surfactant with only one being R<sup>2</sup>. Some alkyl pyridinium salts useful in the present invention have the general formula:

$$\begin{bmatrix} R^2 & N^{+} & X^{-} \\ & & X^{-} \end{bmatrix}$$

wherein R<sup>2</sup> and X- are as defined above. A typical material of this type is cetyl pyridinium chloride.

### Nonionic Surfactant (Alkoxylated Materials)

**[0050]** Suitable nonionic surfactants for use herein include addition products of ethylene oxide and, optionally, propylene oxide, with fatty alcohols, fatty acids, fatty amines, etc.

5 Suitable compounds are substantially water-soluble surfactants of the general formula:

$$R^2 - Y - (C_2H_4O)_7 - C_2H_4OH$$

wherein R<sup>2</sup> is selected from primary, secondary and branched chain alkyl and/or acyl hydrocarbyl groups; primary, secondary and branched chain alkenyl hydrocarbyl groups; and primary, secondary and branched chain alkyl- and alkenyl-substituted phenolic hydrocarbyl groups; said hydrocarbyl groups having a hydrocarbyl chain length of from 8 to 20, preferably from 10 to 18 carbon atoms.

Y is typically -O-, -C(O)O-, -C(O)N(R)-, or -C(O)N(R)R-, in which  $R^2$  and R, when present, have the meanings given hereinbefore, and/or R can be hydrogen, and z is at least 8, preferably at least 10-11.

The nonionic surfactants herein are characterized by an HLB (hydrophilic-lipophilic balance) of from 7 to 20, preferably from 8 to 15.

Examples of particularly suitable nonionic surfactants include

- Straight-Chain, Primary Alcohol Alkoxylates such as tallow alcohol-EO(11), tallow alcohol-EO(18), and tallow alcohol-EO(25);
- Straight-Chain, Secondary Alcohol Alkoxylates such as 2-C<sub>16</sub>EO(11); 2-C<sub>20</sub>EO(11); and 2-C<sub>16</sub>EO(14);
- Alkyl Phenol Alkoxylates, such as p-tridecylphenol EO(11) and p-pentadecylphenol EO(18), as well as
- Olefinic Alkoxylates, and Branched Chain Alkoxylates such as branched chain primary and secondary alcohols which are available from the well-known "OXO" process.

#### **Amine Oxides**

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[0051] Suitable amine oxides include those with one alkyl or hydroxyalkyl moiety of 8 to 28 carbon atoms, preferably from 8 to 16 carbon atoms, and two alkyl moieties selected from alkyl groups and hydroxyalkyl groups with 1 to 3 carbon atoms. Examples include dimethyloctylamine oxide, diethyldecylamine oxide, bis-(2-hydroxyethyl)dodecylamine oxide, dimethyldodecyl-amine oxide, dipropyltetradecylamine oxide, methylethylhexadecylamine oxide, dimethyl-2-hydroxyoctadecylamine oxide, and coconut fatty alkyl dimethylamine oxide.

### Fatty Acids

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[0052] Suitable fatty acids include those containing from 10 to 25, preferably from 12 to 25 total carbon atoms, with the fatty moiety containing from 10 to 22, preferably from 16 to 22, carbon atoms. The shorter moiety contains from 1 to 4, preferably from 1 to 2 carbon atoms. The level of unsaturation of the tallow chain can be measured by the lodine Value (IV) of the corresponding fatty acid, which in the present case should preferably be in the range of from 5 to 100, more preferably in the range of from 0 to 25.

Specific examples of fatty acid compounds suitable for use in the aqueous fabric softening compositions herein include compounds selected from lauric acid, myristic acid, palmitic acid, stearic acid, arachidic acid, behenic acid, oleic acid, coconut fatty acid, tallow fatty acid, partially hydrogenated tallow fatty acid and mixtures thereof. A most preferred fatty acid compound is tallow fatty acid with an lodine Value (IV) of 18.

[0053] Inorganic viscosity/dispersibility control agents which can also act like or augment the effect of the surfactant concentration aids, include water-soluble, ionizable salts which can also optionally be incorporated into the compositions of the present invention. A wide variety of ionizable salts can be used. Examples of suitable salts are the halides of the Group IA and IIA metals of the Periodic Table of the Elements, e.g., calcium chloride, magnesium chloride, sodium chloride, potassium bromide, and lithium chloride. The ionizable salts are particularly useful during the process of mixing the ingredients to make the compositions herein, and later to obtain the desired viscosity. The amount of ionizable salts used depends on the amount of active ingredients used in the compositions and can be adjusted according to the desires of the formulator. Typical levels of salts used to control the composition viscosity are from about 20 to about 20,000 parts per million (ppm), preferably from about 20 to about 11,000 ppm, by weight of the composition.

Alkylene polyammonium salts can be incorporated into the composition to give viscosity control in addition to or in place of the water-soluble, ionizable salts above. In addition, these agents can act as scavengers, forming ion pairs with anionic detergent carried over from the main wash, in the rinse, and on the fabrics, and may improve softness performance. These agents may stabilize the viscosity over a broader range of temperature, especially at low temperatures, compared to the inorganic electrolytes.

Specific examples of alkylene polyammonium salts include I-lysine monohydrochloride and 1,5-diammonium 2-methyl pentane dihydrochloride.

### 4. Other ingredients

[0054] Still other optional ingredients include, but are not limited to enzymes, Soil Release Agents, perfumes, preservatives/stabilisers, chelants, bacteriocides, colorants, optical brighteners, antifoam agents, non-cellulose reactive dye fixing agent such as polyquaternary ammonium compounds (e.g. Sandofix WE56 commercially available from Hoechst, or Rewin SFR commercially available from CHT R. Beitlich), polyamino functional polymer such as disclosed in copending application EP 97201488.0, dispersible polyolefin such as Velustrol® as disclosed in co-pending application PCT/US 97/01644, and the like.

### **Enzymes**

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[0055] The compositions herein can optionally employ one or more enzymes such as lipases, proteases, cellulase, amylases and peroxidases. A preferred enzyme for use herein is a cellulase enzyme. Indeed, this type of enzyme will further provide a color care benefit to the treated fabric. Cellulases usable herein include both bacterial and fungal types, preferably having a pH optimum between 5 and 9.5. U.S. 4,435,307 discloses suitable fungal cellulases from Humicola insolens or Humicola strain DSM1800 or a cellulase 212-producing fungus belonging to the genus Aeromonas, and cellulase extracted from the hepatopancreas of a marine mollusk, Dolabella Auricula Solander. Suitable cellulases are also disclosed in GB-A-2.075.028; GB-A-2.095.275 and DE-OS-2.247.832. CAREZYME® and CELLUZYME® (Novo) are especially useful. Other suitable cellulases are also disclosed in WO 91/17243 to Novo, WO 96/34092, WO 96/34945 and EP-A-0,739,982. In practical terms for current commercial preparations, typical amounts are up to 5 mg by weight, more typically 0.01 mg to 3 mg, of active enzyme per gram of the detergent composition. Stated otherwise, the compositions herein will typically comprise from 0.001% to 5%, preferably 0.01%-1% by weight of a commercial enzyme preparation. In the particular cases where activity of the enzyme preparation can be defined otherwise such as with cellulases, corresponding activity units are preferred (e.g. CEVU or cellulase Equivalent Viscosity Units). For instance, the compositions of the present invention can contain cellulase enzymes at a level equivalent to an activity from about 0.5 to 1000 CEVU/gram of composition. Cellulase enzyme preparations used for the purpose of formulating the compositions of this invention typically have an activity comprised between 1,000 and 10,000 CEVU/gram in liquid form, around 1,000 CEVU/gram in solid form.

### Soil Release Agents

[0056] Soil Release agents are desirably used in fabric softening compositions of the instant invention. Suitable soil release agents include those of U.S. 4,968,451, November 6, 1990 to J.J. Scheibel and E.P. Gosselink: such ester oligomers can be prepared by (a) ethoxylating allyl alcohol, (b) reacting the product of (a) with dimethyl terephthalate ("DMT") and 1,2-propylene glycol ("PG") in a two-stage transesterification/oligomerization procedure and (c) reacting the product of (b) with sodium metabisulfite in water; the nonionic end-capped 1,2-propylene/polyoxyethylene terephthalate polyesters of U.S. 4,711,730, December 8, 1987 to Gosselink et al, for example those produced by transesterification/oligomerization of poly(ethyleneglycol) methyl ether, DMT, PG and poly(ethyleneglycol) ("PEG"); the partly- and fully- anionic-end-capped oligomeric esters of U.S. 4,721,580, January 26, 1988 to Gosselink, such as oligomers from ethylene glycol ("EG"), PG, DMT and Na-3,6-dioxa-8-hydroxyoctanesulfonate; the nonionic-capped block polyester oligomeric compounds of U.S. 4,702,857, October 27, 1987 to Gosselink, for example produced from DMT, Me-capped PEG and EG and/or PG, or a combination of DMT, EG and/or PG, Me-capped PEG and Na-dimethyl-5-sulfoisophthalate; and the anionic, especially sulfoaroyl, end-capped terephthalate esters of U.S. 4,877,896, October 31, 1989 to Maldonado, Gosselink et al, the latter being typical of SRA's useful in both laundry and fabric conditioning products, an example being an ester composition made from m-sulfobenzoic acid monosodium salt, PG and DMT optionally but preferably further comprising added PEG, e.g., PEG 3400. Another preferred soil release agent is a sulfonated end-capped type described in US 5,415,807.

### **Perfumes**

[0057] Perfume can also be incorporated into the invention compositions to provide fully-formulated fragrance, and thus, desirable in-package and in-use (wash-time) fragrance.

[0058] In formulating the present fabric softening compositions, the fully-formulated fragrance can be prepared using numerous known odorant ingredients of natural or synthetic origin. The range of the natural raw substances can embrace not only readily-volatile, but also moderately-volatile and slightly-volatile components and that of the synthet-

ics can include representatives from practically all classes of fragrant substances, as will be evident from the following illustrative compilation: natural products, such as tree moss absolute, basil oil, citrus fruit oils (such as bergamot oil, mandarin oil, etc.), mastix absolute, myrtle oil, palmarosa oil, patchouli oil, petitgrain oil Paraguay, wormwood oil, alcohols, such as farnesol, geraniol, linalool, nerol, phenylethyl alcohol, rhodinol, cinnamic alcohol, aldehydes, such as cit-Lilial™ alpha-hexyl-cinnamaldehyde, hydroxycitronellal, ral, (p-tert.butyl-alpha methyldihydrocinnamaldehyde), methylnonylacetaldehyde, ketones, such as allylionone, alpha-ionone, beta -ionone, isoraldein (isomethyl- alpha -ionone), methylionone, esters, such as allyl phenoxyacetate, benzyl salicylate, cinnamyl propionate, citronellyl acetate, citronellyl ethoxolate, decyl acetate, dimethylbenzylcarbinyl acetate, dimethylbenzylcarbinyl butyrate, ethyl acetoacetate, ethyl acetylacetate, hexenyl isobutyrate, linalyl acetate, methyl dihydrojasmonate, styrallyl acetate, vetiveryl acetate, etc., lactones, such as gamma-undecalactone, various components often used in perfumery, such as musk ketone, indole, p-menthane-8-thiol-3-one, and methyl-eugenol. Likewise, any conventional fragrant acetal or ketal known in the art can be added to the present composition as an optional component of the conventionally formulated perfume (c). Such conventional fragrant acetals and ketals include the well-known methyl and ethyl acetals and ketals, as well as acetals or ketals based on benzaldehyde, those comprising phenylethyl moieties, or more recently developed specialities such as those described in a United States Patent entitled "Acetals and Ketals of Oxo-Tetralins and Oxo-Indanes, see U.S. Pat. No. 5 ,084,440, issued January 28, 1992, assigned to Givaudan Corp. Of course, other recent synthetic specialities can be included in the perfume compositions for fully-formulated fabric softening compositions. These include the enol ethers of alkyl-substituted oxo-tetralins and oxo-indanes as described in U.S. Pat. 5,332,725, July 26, 1994, assigned to Givaudan; or Schiff Bases as described in U.S. Pat. 5,264,615, December 9, 1991, assigned to Givaudan. It is preferred that the pro-fragrant material be added separately from the conventional fragrances to the fabric softening compositions of the invention.

### **Stabilizers**

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[0059] Stabilizers can be present in the compositions of the present invention. The term "stabilizer," as used herein, includes antioxidants and reductive agents. These agents are present at a level of from 0% to about 2%, preferably from about 0.01% to about 0.2%, more preferably from about 0.035% to about 0.1% for antioxidants, and more preferably from about 0.01% to about 0.2% for reductive agents. These assure good odor stability under long term storage conditions for the compositions and compounds stored in molten form. The use of antioxidants and reductive agent stabilizers is especially critical for low scent products (low perfume).

Examples of antioxidants that can be added to the compositions of this invention include a mixture of ascorbic acid, ascorbic palmitate, propyl gallate, available from Eastman Chemical Products, Inc., under the trade names Tenox® PG and Tenox S-1; a mixture of BHT (butylated hydroxytoluene), BHA (butylated hydroxyanisole), propyl gallate, and citric acid, available from Eastman Chemical Products, Inc., under the trade name Tenox-6; butylated hydroxytoluene, available from UOP Process Division under the trade name Sustane® BHT; tertiary butylhydroquinone, Eastman Chemical Products, Inc., as Tenox TBHQ; natural tocopherols, Eastman Chemical Products, Inc., as Tenox GT-1/GT-2; and butylated hydroxyanisole, Eastman Chemical Products, Inc., as BHA; long chain esters (C<sub>8</sub>-C<sub>22</sub>) of gallic acid, e.g., dodecyl gallate; Irganox® 1010; Irganox® 1035; Irganox® B 1171; Irganox® 1425; Irganox® 3114; Irganox® 3125; and mixtures thereof; preferably Irganox® 3125, Irganox® 1425, Irganox® 3114, and mixtures thereof; more preferably Irganox® 3125 alone or mixed with citric acid and/or other chelators such as isopropyl citrate, Dequest® 2010, available from Monsanto with a chemical name of 1-hydroxyethylidene-1, 1-diphosphonic acid (etidronic acid), and Tiron®, available from Kodak with a chemical name of diethylenetriaminepentaacetic acid. The chemical names and CAS numbers for some of the above stabilizers are listed in Table II below.

TABLE II

	Antioxidant	CAS No.	Chemical Name used in Code of Federal Regulations
50	Irganox <sup>®</sup> 1010	6683-19-8	Tetrakis (methylene(3,5-di-tert-butyl-4 hydroxyhydrocinnamate)) methane
	Irganox <sup>®</sup> 1035	41484-35-9	Thiodiethylene bis(3,5-di-tert-butyl-4-hydroxyhydrocinnamate
	Irganox <sup>®</sup> 1098	23128-74-7	N,N'-Hexamethylene bis(3,5-di-tert-butyl-4-hydroxyhydrocinnamamide
55	Irganox <sup>®</sup> B 1171	31570-04-4	1:1 Blend of Irganox <sup>®</sup> 1098 and
		23128-74-7	Irgafos <sup>®</sup> 168
	Irganox <sup>®</sup> 1425	65140-91-2	Calcium bis(monoethyl(3,5-di-tert-butyl-4-hydroxybenzyl)phosphonate)

### TABLE II (continued)

	Antioxidant	CAS No.	Chemical Name used in Code of Federal Regulations
	Irganox <sup>®</sup> 3114	65140-91-2	Calcium bis(monoethyl(3,5-di-tert-butyl-4-hydroxybenzyl)phosphonate)
i	Irganox <sup>®</sup> 3125	34137-09-2	3,5-Di-tert-butyl-4-hydroxy-hydrocinnamic acid triester with 1,3,5-tris(2-hydroxye-thyl)-S-triazine-2,4,6-(1H, 3H, 5H)-trione
	Irgafos <sup>®</sup> 168	31570-04-4	Tris(2,4-di-tert-butyl-phenyl)phosphite

[0060] Examples of reductive agents include sodium borohydride, hypophosphorous acid, lrgafos<sup>®</sup> 168, and mixtures thereof.

### Form of the composition

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**[0061]** The fabric softening composition can take a variety of physical forms including liquid such as aqueous or non-aqueous compositions and solid forms such as solid particulate forms.

Such compositions may be applied onto a substrate such as a dryer sheet product, used as a rinse added product, or as a spray or foam product.

**[0062]** Accordingly, there is provided a method for the domestic treatment of a fabric to reduce the amount of dye released from the fabric during wet treatments and comprising the step of:

- a)-contacting the fabrics with a cellulose reactive dye fixing agent or a composition thereof as defined herein before; and
- b)- thereafter subjecting the treated fabrics to a heating source, said heating source being provided in a dry medium.

**[0063]** Preferably, the contacting of the fabrics with a cellulose reactive dye fixing agent or a composition incorporating such dye fixing agent occurs during the rinse cycle of a washing process.

Preferably, the cellulose reactive dye fixing agent in the method herein above is incorporated in a fabric softening composition. The resulting fabric softening composition is as described herein before.

**[0064]** Preferred heating source for use herein are those in which a temperature of at least 60°C, more preferably at least 80°C is used, such as those commonly known in domestic processes, e.g. in tumble drying processes, ironing processes or even combination of the above processes.

**[0065]** Dry medium is an important feature of the method aspect of the invention. In contrast, the use of the heating source in an aqueous medium would not provide sufficient covalent linkage of the cellulose reactive dye fixing agent with the dye. It has been found that preferably for the linkage to occur, the presence of water is to be reduced to a minimum, that is to less than the moisture regain content of the fabric being treated.

The moisture regain content is defined as in D2654-89a providing Standard Test Methods for Moisture in Textiles, page 724-733. Of course, the value for the moisture regain content is specific to the relative humidity, ambient temperature and type of fabric. In varying at least one of these characteristics, the moisture regain content value will also vary.

However, what is preferred for the purpose of the invention is that the content of water in the fabric is below its moisture regain content for a good chemical linkage to take place.

**[0066]** Reducing the amount of water on the fabrics to less than the moisture regain content is easily achieved by drying the fabrics in a tumble dryer and/or ironing these dried fabrics.

### **Process**

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**[0067]** The fabric softening composition can conveniently be made according to well known processes to the skilled person. An exemplary disclosure is given in EP-A-0,668,902.

0 [0068] The invention is illustrated in the following non limiting examples, in which all percentages are on a weight basis unless otherwise stated.

[0069] In the examples, the abbreviated component identifications have the following meanings:

DEQA : Di-(tallowyl-oxy-ethyl) dimethyl ammonium chloride DOEQA : Di-(oleyloxyethyl) dimethyl ammonium methylsulfate

DTDMAC : Ditallow dimethylammonium chloride

DHEQA : Di-(soft-tallowyl-oxy-ethyl) hydroxyethyl methyl ammonium methylsulfate

Fatty acid : Tallow fatty acid IV=18

Electrolyte : Calcium chloride

DTDMAMS : Ditallow dimethyl ammonium methylsulfate

SDASA : 1:2 Ratio of stearyldimethyl amine:triple-pressed stearic acid Glycosperse S-20 : Polyethoxylated sorbitan monostearate available from Lonza

Clay : Calcium Bentonite Clay, Bentonite L, sold by Southern Clay Products

TAE25 : Tallow alcohol ethoxylated with 25 moles of ethylene oxide per mole of alcohol

PEG : Polyethylene Glycol 4000

Dye Fix 1 : Cellulose reactive dye fixing agent available under the tradename Indosol CR from Clariant

Dye Fix 2 : Cellulose reactive dye fixing agent available under the tradename Rewin WBS from CHT R.

**Beitlich** 

### Example 1

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[0070] The following compositions according to the invention are in accordance with the invention

Component	Α	В	С	D	E	F
DTDMAC	-	-	-	-	4.5	15
DEQA	2.6	2.9	18.0	19.0	-	-
TAE25		-	1.0	-	-	-
Hydrochloride	0.02	0.02	0.02	0.02	0.02	0.02
acid						
PEG	-	-	0.6	0.6	-	0.6
Perfume	1.0	1.0	1.0	1.0	1.0	1.0
Silicone	0.01	0.01	0.01	0.01	0.01	0.01
antifoam						
Dye fix 1	1.0	0.5	5.0	-	1.0	-
Dye fix 2	-	0.5	-	3.0	-	5.0
Electrolyte (ppm)	-	-	600	600	-	600
Dye (ppm)	10	10	50	50	10	50
Water and minors to balance to 100						

[0071] 160 g of fabrics were treated with composition A. The fabrics were then line dried and then submitted to an ironing process set on linen. It is then observed after a further wash cycle that the fabric treated in this manner exhibit better dye fixing performance than fabrics which have not undergo a ironing process. The same results were obtained with compositions B to F.

### Example 2

[0072] The following compositions for use as dryer-added sheets are in accordance with the invention

	G	Н	I
DOEQA	40	-	-
DHEQA	-	20	-
DTDMAMS	-	-	20

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(continued)

	G	Н	I
SDASA	30	20	30
Glycosperse S-20	-	10	-
Glycerol Monostearate	-	-	20
Clay	4	3	4
Perfume	0.7	0.7	1.6
Dye fix 1	2.0	1.0	2.0
Stearic acid to balance			

### 15 Claims

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- 1. A fabric softening composition comprising a cationic fabric softener having at least two long chains and a cellulose reactive dye fixing agent.
- 20 **2.** A fabric softening composition according to Claim 1, wherein said fabric softener component is a cationic biodegradable fabric softener of formula:

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$$\begin{bmatrix} R^{3} & R^{2} \\ + & N - (CH_{2})_{n} - Q - T \end{bmatrix} X - \begin{bmatrix} R^{3} & R^{3} \\ + & N - (CH_{2})_{n} - CH - CH_{2} \\ R^{3} & Q & Q \\ & T! & T^{2} \end{bmatrix} X$$
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$$(I)$$

$$(II)$$

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wherein Q is selected from -O-C(O)-, -C(O)-O-, -O-C(O)-O-, -NR^4-C(O)-, -C(O)-NR^4-;  $R^1 \text{ is } (CH_2)_n\text{-}Q\text{-}T^2 \text{ or } T^3 \text{ or } R^3; \\ R^2 \text{ is } (CH_2)_m\text{-}Q\text{-}T^4 \text{ or } T^5 \text{ or } R^3; \\ R^3 \text{ is } C_1\text{-}C_4 \text{ alkyl or } C_1\text{-}C_4 \text{ hydroxyalkyl or } H; \\ R^4 \text{ is H or } C_1\text{-}C_4 \text{ alkyl or } C_1\text{-}C_4 \text{ hydroxyalkyl;} \\ T^1, T^2, T^3, T^4, T^5 \text{ are independently } C_{11}\text{-}C_{22} \text{ alkyl or alkenyl;} \\ n \text{ and m are integers from 1 to 4; and}$ 

45 X is a softener-compatible anion.

- 3. A fabric softening composition according to Claim 2, wherein said fabric softener is present in amount of 1% to 80% by weight.
- 4. A fabric softening composition according to any one of Claims 1-3, wherein said cellulose reactive dye fixing agent is a product containing the reactive group of the reactive dye classes selected from halogeno-triazine products, vinyl sulphones compounds, epichlorhydrine derivatives, hydroxyethylene urea derivatives, formaldehyde condensation products, polycarboxylates, glyoxal and glutaraldehyde derivatives and mixtures thereof.
- 55 5. A fabric softening composition according to Claim 4, wherein said cellulose reactive dye fixing agent is a formaldehyde condensation product selected from the condensation products derived from formaldehyde and a group selected from an amino-group, an imino-group, a phenol group, an urea group, a cyanamide group and an aromatic group.

- **6.** A fabric softening composition according to Claim 5, wherein said cellulose reactive dye fixing agent is present in amount of 0.01% to 50% by weight, preferably from 0.01% to 25% by weight of the composition.
- 7. A method for the domestic treatment of a fabric to reduce the amount of dye released from the fabric during wet treatments and comprising the step of: a)-contacting the fabrics with a cellulose reactive dye fixing agent; and b)-thereafter subjecting the treated fabrics to a heating source in a dry medium.

- 8. A method for the domestic treatment of a fabric to reduce the amount of dye released from the fabric during wet treatments and comprising the step of: a)-contacting the fabrics with a fabric softening composition as defined in any one of Claims 1-6; and b)- thereafter subjecting the treated fabrics to a heating source in a dry medium.
- **9.** A method according to either one of Claim 7 or 8, wherein said heating source is provided by a process selected from a tumble-drying process, ironing process and mixtures thereof.



# **EUROPEAN SEARCH REPORT**

Application Number

EP 97 87 0186

Category	Citation of document with indicat of relevant passages	on, where appropriate,	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
(	WO 96 27649 A (PROCTER * page 3, paragraph 3; * page 6, line 1 - page	claims $1,3,5-7 *$	1-6	C11D3/00 C11D1/62
Х	WO 94 04643 A (COLGATE * claims 1-6; examples		1-4,6	
A	DE 31 05 897 A (SANDOZ * example 23 *	AG) 	1-9	
				TECHNICAL FIELDS SEARCHED (Int.Cl.6)
				C11D
	The present search report has been			
	Place of search	Date of completion of the search	<u> </u>	Examiner
	THE HAGUE	8 April 1998		selet-Taisne, S
X : part Y : part	ATEGORY OF CITED DOCUMENTS  icularly relevant if taken alone icularly relevant if combined with another ument of the same category	E : earlier paten after the filing	nciple underlying the t document, but publi g date ted in the application ed for other reasons	