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(54) **Stable fabric conditioning composition**

(57) An aqueous fabric conditioning composition containing a biodegradable quaternary ammonium softening agent containing at least one ester group wherein the composition has a pH below 2.0 The fabric conditioning composition is stable at room temperature and has excellent resistance to microbiological contamination.

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FABRIC SOFTENING COMPOSITIONTECHNICAL FIELD

5 The present invention relates to fabric softening
compositions, in particular the invention relates to aqueous
dispersions of biodegradable fabric softening compositions
comprising a water insoluble cationic fabric softening agent
10 having excellent hydrolytic stability and microbial
resistance.

BACKGROUND OF INVENTION

15 Rinse added fabric softener compositions are well known,
however one of the problems associated with fabric softening
compositions is the physical and chemical instability of such
compositions when stored. This problem is accentuated by
having a concentrated composition. EP 0 239 910 (Procter &
Gamble) attempts to overcome the problem by specifying that
20 rinse conditioners containing specific biodegradable
quaternary ammonium compounds that fabric conditioners should
have a pH ranging between 2.5 to 4.2. EP 0 239 910 states
that a pH outside this range does not have such excellent
hydrolytic stability on prolonged storage.

25 A further problem associated with rinse conditioners is that
of microbiological contamination. Many of the rinse
conditioners that are for sale on the commercial market have
antimicrobial ingredients to prevent microbiological growth
30 within the compositions.

The present invention solves the technical problem of
providing a hydrolytically stable rinse conditioner with
excellent resistance to microbiological contamination without
35 the need for conventional antimicrobial ingredients.

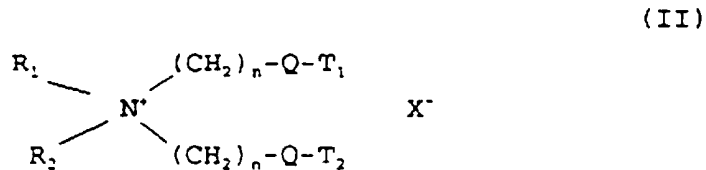
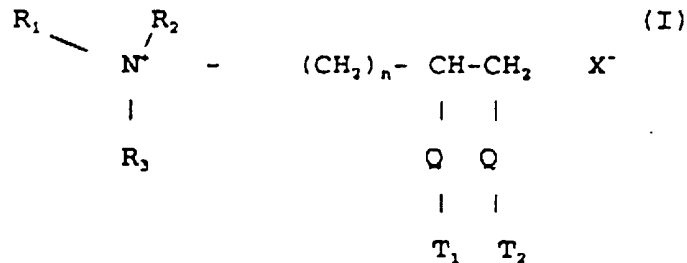
DESCRIPTION OF INVENTION

Accordingly the present invention provides an aqueous fabric conditioning composition containing a biodegradable quaternary ammonium softening agent containing at least one ester group wherein the composition has a pH below 2.0

According to another aspect the invention provides the use of a fabric conditioner having a pH less than 2 to enhance the effect a soil release polymer within a rinse conditioner.

DETAILED DESCRIPTION OF INVENTIONQuaternary Ammonium Softening Compound

It is preferred if the quaternary ammonium softening compound is of the formula I or formula II:



in which Q is $\begin{array}{c} \text{O} \\ || \\ -\text{O}-\text{C}- \end{array}$ or $\begin{array}{c} \text{O} \\ || \\ -\text{C}-\text{O} \end{array}$ or $\begin{array}{c} \text{O} \\ || \\ \text{O}-\text{C}-\text{O} \end{array}$,

R₁, R₂, R₃ are independently selected from C₁-C₄ alkyl or C₂-C₄ alkenyl;

T_1 , T_2 are independently selected from C_{12} - C_{22} alkyl or alkenyl chains, n is a number between 0 and 4.

5 X^- is a softener compatible anion

Especially preferred materials of this class and their method of preparation are, for example, described in US 4 137 180 (Lever Brothers). Preferably these materials comprise small
10 amounts of the corresponding monoester as described in US 4 137 180 for example 1-tallowoxy, 2-hydroxytrimethyl ammonium propane chloride. It is particularly advantageous if the rinse softening agent is 1,2 bis [hardened tallowoyl-oxy-
15 ethyl] N,N-trimethyl ammonium chloride.

A second particularly preferred quaternary ammonium softening agent is N,N-di(tallowoyl-oxy-ethyl) N,N-dimethyl
20 ammonium chloride.

Preferably the level of ester linked quaternary ammonium compounds is at least 1% by weight of the composition, more
25 preferably more than 3% by weight of the composition; especially interesting are concentrated compositions which comprise more than 7% of ester-linked quaternary ammonium compound. The level of ester-linked quaternary ammonium compounds preferably is between 1% and 80% by weight, more
30 preferably 2% to 50%, most preferably 8% to 30%.

Soil Release Agent

Optionally, the composition herein contain from 0.1% to 10%, preferably 0.2% to 5%, of a soil release agent. Preferably, such a soil release agent of the present composition is a polymer. Polymeric soil release agents useful in the present invention include hydroxyether cellulosic polymers, copolymeric blocks of terephthalate and polyethylene oxide or polypropylene oxide, and cationic guar gums, and the like.

The cellulosic derivatives that are functional as soil release agents may be characterised as certain hydroxyethers of cellulose such as Methocel® (Dow); also, certain cationic cellulose ether derivatives such as Polymer JR-125^R, JR-400^R, and JR-30M^R (Union Carbide).

Other effective soil release agents are cationic guar gums such as Jaguar Plus^R (Stein Hall) and Gendrive 458^R (General Mills).

A preferred fabric conditioning composition has a polymeric soil release agent selected from the group consisting of methyl cellulose, hydroxypropyl methylcellulose, or hydroxybutyl methylcellulose, said cellulosic polymer having a viscosity in 2% aqueous solution at 20°C of $15 \cdot 10^{-3}$ to 75 Pa.s (15 to 75,000 centipoise).

A more preferred soil release agent is a copolymer having blocks of terephthalate and polyethylene oxide. More specifically, these polymers are comprised of repeating units of ethylene terephthalate and polyethylene oxide terephthalate at a molar ratio of ethylene terephthalate units to polyethylene oxide terephthalate units of from about 25:75 to about 35:65, said polyethylene oxide terephthalate

containing polyethylene oxide blocks having molecular weight of from about 300 to about 2000. The molecular weight of this polymeric soil release agent is in the range of from about 5,000 to about 55,000.

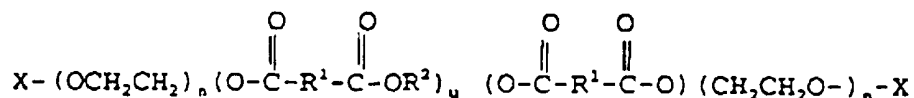
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Another preferred polymeric soil release agent is a crystallizable polyester with repeat units of ethylene terephthalate units containing 10-15% by weight of ethylene terephthalate units together with 90-50% by weight of polyoxyethylene terephthalate units, derived from a polyoxyethylene glycol of average molecular weight 300-6,000, and the molar ratio of ethylene terephthalate units to polyoxyethylene terephthalate units in the crystallizable polymeric compound is between 2:1 and 6:1. Examples of this polymer include the commercially available material Zelcon[®] 4780 (from Dupont) and Milease[®]T (from ICI).

15

Highly preferred soil release agents are polymers of the generic formula:

20



25

in which X can be any suitable capping group, with each X being selected from the group consisting of H, and alkyl or acyl groups containing from 1 to about 4 carbon atoms. n is selected for water solubility and generally is from about 6 to about 113, preferably from about 10 to about 50. u is critical to formulation in a liquid composition having a relatively high ionic strength. There should be very little material in which u is greater than 10. Furthermore there should be at least 20%, preferably at least 40% of material in which u ranges from 3 to 5.

35

The R¹ moieties are essentially 1,4-phenylene moieties. As used herein, the term "the R¹ moieties consist entirely of 1,4-phenylene moieties, or are partially substituted with other arylene or alkarylene moieties, alkylene moieties, alkenylene moieties, or mixtures thereof. Arylene and alkarylene moieties which can be partially substituted for 1,4-phenylene include 1,3-phenylene, 1,2-phenylene, 1,8-naphtylene, 1,4-naphtylene, 2,2-biphenylene, 4,4-biphenylene and mixtures thereof. Alkylene and alkenylene moieties which can be partially substituted include ethylene, 1,2-propylene, 1,4-butylene, 1,5-pentylene, 1,6-hexamethylene, 1,7-heptamethylene, 1,8-octamethylene, 1,4-cyclohexylene, and mixtures thereof.

For the R¹ moieties, the degree of partial substitution with moieties other than 1,4-phenylene should be such that the soil release properties of the compound are not adversely affected to any great extent. Generally, the degree of partial substitution which can be tolerated will depend upon the backbone length of the compound, i.e., longer backbones can have greater partial substitution for 1,4-phenylene moieties. Usually, compounds where the R¹ comprise from about 50 to 100% 1,4-phenylene moieties (from 0 to about 50% moieties other than 1,4-phenylene) have adequate soil release activity. For example, polyesters made according to the present invention with a 40:60 mole ratio of isophthalic (1,3-phenylene) to terephthalic (1,4-phenylene) acid have adequate soil release activity. However, because most polyesters used in fiber making comprise ethylene terephthalate units, it is usually desirable to minimize the degree of partial substitution with moieties other than 1,4-phenylene for best soil release activity. Preferably, the R¹ moieties consist entirely of (i.e., comprise 100%) 1,4-phenylene moieties, i.e. each R¹ moiety is 1,4-phenylene. (Irrespective of the mechanism of action, it is surprising that the soil release

polymers do show excellent benefits on fabrics other than polyester fabrics and the compositions herein are designed to clean all manner of fabrics and textiles.)

5

For the R^2 moieties, suitable ethylen or substituted ethylene moieties include ethylene, 1,2-propylene, 1,2-butylene, 1,2-hexylene, 3-methoxy-1,2-propylene and mixtures thereof.

10

Preferably, the R^2 moieties are essentially ethylene moieties, 1,2-propylene moieties or mixture thereof.

Inclusion of a greater percentage of ethylene moieties tends to improve the soil release activity of the compounds.

Surprisingly, inclusion of a greater percentage of 1,2-propylene moieties tends to improve the water solubility of the compounds.

15

For this invention, the use of 1,2-propylene moieties or a similar branched equivalent is desirable for incorporation of any substantial part of the soil release component in the liquid fabric softener compositions. Preferably, from about 75% to about 100%, more preferably from about 90% to about 100% of the R^2 moieties are 1,2-propylene moieties.

20

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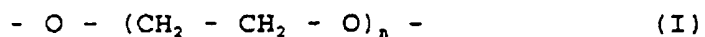
The value for each n is at least about 6, but is preferably at least about 10. The value for each n usually ranges from about 12 to about 113. Typically, the value for each n is in the range of from about 12 to about 43.

30

A second type of preferred soil release polymers are polymers which comprises a copolymer having:

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- (i) monomer units of poly(ethylene glycol) and/or capped poly(ethylene glycol) having the formula I



5

and/or the formula Ia:

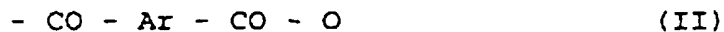


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wherein X is a hydrogen atom or a capping group, preferably a C₁₋₄ alkyl group; R is a hydrogen atom or a methyl group; and n is an integer;

15

- (ii) monomer units of an aromatic dicarboxylic acid, having the formula II:

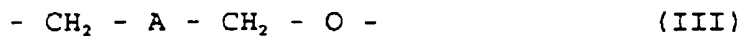


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wherein Ar is a bifunctional aromatic group; and

- (iii) monomer units of a polyol having at least 3 hydroxyl groups, having the formula III:

25



wherein A is a bifunctional group containing at least 1 carbon atom and at least 1 hydroxyl group.

30

35

Other Ingredients

The composition can also contain nonionic stabilisers.

Suitable nonionic stabilisers which can be used include the
5 condensation products of $C_8 - C_{20}$ primary or secondary
predominantly linear alcohols with 10 to 20 moles of ethylene
oxide. The alcohols may be saturated or unsaturated. In
particular Genapol T-110, Genapol T-150, Genapol T-200,
Genapol C-200 all ex Hoeschst AG, Lutensol AT18 ex BASF,
10 Genapol 0-100 and Genapol 0-150 ex Hoechst, or fatty alcohols
for example Laurex CS, ex Albright and Wilson or Adol 340 ex
Sherex. Preferably the nonionic stabiliser has an HLB of
between 10 and 25, more preferably 12 and 20.

15 Preferably, the level of nonionic stabiliser is within the
range from 0.1 to 10% by weight, more preferably from 0.5 to
5% by weight, most preferably from 1 to 4% by weight. The
mole ratio of the quaternary ammonium compound to the
20 nonionic stabilising agent is within the range from 40:1 to
about 1:1, preferably within the range from 18:1 to about
3:1.

25 The composition can also contain fatty acids for example
 $C_8 - C_{24}$ alkyl or alkenyl monocarboxylic acids or polymers
thereof. Preferably saturated fatty acids are used, in
particular, hardened tallow $C_{16} - C_{18}$ fatty acids. Preferably
the fatty acid is non-saponified, more preferably the fatty
30 acid is free for example oleic acid, lauric acid or tallow
fatty acid.

The level of fatty acid material is preferably more than 0.1% by weight, more preferably more than 0.2% by weight. Especially preferred are concentrates comprising from 0.5 to 20% by weight of fatty acid, more preferably 1% to 10% by weight. The weight ratio of quaternary ammonium material to fatty acid material is preferably from 10:1 to 1:10.

The composition can also contain one or more optional ingredients, selected from non-aqueous solvents, pH buffering agents, perfumes, perfume carriers fluorescers colorants, hydrotropes, antifoaming agents, antiredeposition agents, enzymes optical brightening agents, opacifiers, anti-shrinking agents, anti-wrinkle agents, anti-spotting agents, germicides, fungicides, anti-oxidants, anti-corrosion agents, drape imparting agents, antistatic agents and ironing aids.

The composition may also contain nonionic fabric softening agents such as lanolin and derivatives thereof.

EXAMPLES

The invention will now be described with reference to the following non-limiting Examples.

Examples of the invention are illustrated by reference to a number, whilst comparative Examples are described with reference to a letter.

Preparation of ExamplesExample A

5 HT-TMAPC was melted and mixed, with vigourous stirring, with
a solution, at 70°C, comprising demineralised water and
Genapol C100*. After stirring for 10 minutes, the
formulation was allowed to cool to room temperature and then
re-weighed. The sample weight was further diluted with
10 additional demineralised water (thoroughly stirred in).
Sample pH: 3.03.

Example A had the following composition:

15 4.5% HT-TMAPC
0.4% HTFA
0.1% nonionic - Genapol C100*.

20 Genapol C100* is coco-alcohol ethoxylated with 20 ethoxy
units per molecule.

25 HT-TMAPC is 1,2 bis [hardened tallowoyl-oxy-ethyl] N,N
dimethyl ammonium chloride.

Examples 1-4

30 1N hydrochloric acid was added to samples of Example A with
stirring, to give thoroughly mixed formulations having the
following acid levels and pH values:

35

Table 1

| Example | Original Sample Weight (gm) | 1N HCl added (ml) | %HCl | pH |
|---------|-----------------------------|-------------------|------|------|
| 1 | 62.02 | 0.31 | 0.02 | 2.48 |
| 2 | 62.06 | 0.82 | 0.05 | 2.03 |
| 3 | 62.20 | 2.70 | 0.15 | 1.53 |
| 4 | 62.32 | 9.37 | 0.48 | 1.07 |

Hydrolytic Stability

The hydrolytic stability of Example 1-4 and Examples A was monitored by measuring the level of fatty acid in the formulations. This was done by potentiometric titration with tetrabutylammonium hydroxide in isopropanol. The results for samples stored at 20°C and 37°C are presented below. Mean values are of two or three independent samples. The results are shown in Tables 2 and 3.

Table 2

| Example | pH | % Fatty Acid - 37°C mean, [standard dev.] | | | % Fatty Acid - 20°C mean, [standard dev.] | | |
|---------|-----|--|------------|--|--|------------|----------|
| | | 3 weeks | 6 weeks | | 3 weeks | 6 weeks | 36 weeks |
| A | 3 | 0.85[0.02] | 1.01[0.03] | | 0.78[0.02] | 0.93[0.04] | 0.92 |
| 1 | 2.5 | 0.89[0.05] | 1.04[0.08] | | 0.75[0.04] | 0.80[0.04] | 0.82 |
| 2 | 2 | 0.80[0.04] | 1.05[0.03] | | 0.77[0.04] | 0.85[0.00] | 0.77 |
| 3 | 1.5 | 0.83[0.02] | 0.94[0.01] | | 0.80[0.02] | 0.87[0.06] | 0.82 |
| 4 | 1 | ----- | ----- | | 0.89[0.02] | 0.91[0.01] | 1.10 |

As can be seen, the pH 2.5-1.5 samples do not undergo more hydrolysis than the control sample and even the pH 1 sample has acceptable hydrolytic stability when compared to the control. The pH stability of the samples was also very good, as shown in table 3.

Table 3

| Example | pH | pH after 37°C storage | | pH after 20°C storage | |
|---------|---------|-----------------------|---------|-----------------------|---------|
| | Initial | 3 weeks | 6 weeks | 3 weeks | 6 weeks |
| A | 3.03 | 2.94 | 2.86 | 3.08 | 2.99 |
| 1 | 2.48 | 2.47 | 2.39 | 2.52 | 2.43 |
| 2 | 2.03 | 2.04 | 1.95 | 2.07 | 1.97 |
| 3 | 1.53 | 1.55 | 1.43 | 1.55 | 1.46 |
| 4 | 1.07 | 1.05 | 0.99 | 1.11 | 1.02 |

Example B

The following formulation was prepared in analogous manner to Example 1:

14.2% DEEDMAC*

0.3% hardened tallow fatty acid

0.9% perfume

0.05% Hydrochloric Acid

This sample was found to have a pH of 2.18.

DEEDMAC, ex Hoechst di(hardened tallowoxyloxyethyl)dimethyl ammonium chloride: 85% ad., also containing HTFA at 2%, and 13% solvent.

Example 5

5 To 200g of the formulation of Example 6 was added 5.2g of 1N orthophosphoric acid to give a sample containing 0.25% phosphoric acid. After thorough mixing this sample was found to have a pH of 1.76.

Example 6

10 To a further 200g of the formulation of Example 6 was added 7.765g of 85% orthophosphoric acid to give a sample containing 3.18% phosphoric acid. After thorough mixing this sample was found to have a pH of 1.20.

15 The ambient temperature hydrolytic stability of Example B and Examples 5-6 was monitored by measuring the ester quaternary level by two-phase compleximetric titration with SDS. The results given below again show surprisingly good stability for the low pH samples. (The lower initial quat. levels for
20 the lower pH samples is a result of "dilution" with the aqueous phosphoric acid.)

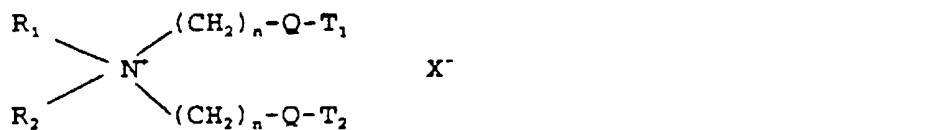
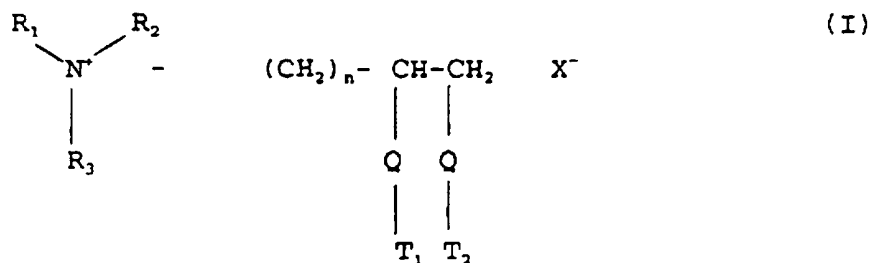
| Example | pH | % Ester Quat. | | | |
|---------|-----|---------------|---------|---------|---------|
| | | Initial | 2 weeks | 4 weeks | 8 weeks |
| B | 2.2 | 14.2 | 14.3 | 14.4 | 14.2 |
| 5 | 1.8 | 13.9 | 14.1 | 13.9 | 13.7 |
| 6 | 1.2 | 13.8 | 13.4 | 13.1 | 12.2 |

25

CLAIMS

1. An aqueous fabric conditioning composition containing a biodegradable quaternary ammonium softening agent containing at least one ester group wherein the composition has a pH below 2.0

2. An aqueous fabric conditioning composition according to claim 1 in which the biodegradable quaternary ammonium softening agent of the formula I or formula II:



in which Q is $-O-\overset{\overset{O}{\parallel}}{C}-$ or $-\overset{\overset{O}{\parallel}}{C}-O$ or $O-\overset{\overset{O}{\parallel}}{C}-O$:

R_1, R_2, R_3 are independently selected from C_1-C_4 alkyl or C_2-C_4 alkenyl;

T_1, T_2 are independently selected from $C_{12}-C_{22}$ alkyl or alkenyl chains, n is a number between 0 and 4.

X^- is a softener compatible anion

3. An aqueous fabric conditioning composition according to claim 1 or claim 2 in which the pH is less than 1.5.

5 4. An aqueous fabric conditioning composition according to any preceding claim in which the composition further comprises a soil release agent.

10 5. An aqueous fabric conditioning composition according to any preceding claim in which the quaternary ammonium softening agent is 1,2 bis [hardened tallowoyl-oxy-ethyl] N,N-trimethyl ammonium chloride.

15 6. An aqueous fabric conditioning composition according to any one of claims 1 to 4 in which the quaternary ammonium softening agent is N,N-di(tallowoyl-oxy-ethyl) N,N-dimethyl ammonium chloride.

20 7. An aqueous fabric conditioning composition according to any preceding claim in which the level of quaternary ammonium softening agent is from 2 wt% to 50 wt%.

25 8. Use of a fabric conditioner having a pH less than 2 to enhance the effect a soil release polymer within a rinse conditioner.



Application No: GB 9609745.6
Claims searched: 1-7

Examiner: Peter Davey
Date of search: 5 November 1996

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.O): D1P (PDP, PDX, PE, PFF, PH)

Int Cl (Ed.6): D06M 13/46 13/463 13/467 13/47 13/473 13/477

Other: Online: WPI

Documents considered to be relevant:

| Category | Identity of document and relevant passage | Relevant to claims |
|----------|--|--------------------|
| X | GB 2188653 A (PROCTER & GAMBLE), see eg. page 1, lines 24-26 and page 3, lines 1-6 | 1 at least |
| X | EP 0409504 A2 (UNILEVER), see eg. page 4, lines 2-4 and page 6, lines 14-16 | 1 at least |
| X | EP 0409502 A2 (UNILEVER), see eg. page 2, lines 8-10 and page 3, lines 37-38 | 1 at least |
| X | WPI Abstract Acc. No. 93-165029/20 and JP 050098571 A (LION CORPN.), see abstract | 1 at least |

| | | | |
|---|---|---|--|
| X | Document indicating lack of novelty or inventive step | A | Document indicating technological background and/or state of the art. |
| Y | Document indicating lack of inventive step if combined with one or more other documents of same category. | P | Document published on or after the declared priority date but before the filing date of this invention. |
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