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(71) Applicant : **UNILEVER PLC**
Unilever House Blackfriars P.O. Box 68
London EC4P 4BQ (GB)
(84) **GB**

(71) Applicant : **UNILEVER N.V.**
Burgemeester s'Jacobplein 1 P.O. Box 760
NL-3000 DK Rotterdam (NL)
(84) **CH DE ES FR IT LI NL SE**

(72) Inventor : **Ellis, Simon Richard, Unilever Research**
Port Sunlight Laboratory, Quarry Road East
Bebington, Wirral, Merseyside L63 3JW (GB)
Inventor : **Turner, Graham Andrew Unilever Research**
Port Sunlight Laboratory, Quarry Road East
Bebington, Wirral, Merseyside L63 3JW (GB)

(74) Representative : **Bristow, Stephen Robert et al**
Unilever PLC Patent Division Colworth House
Sharnbrook
Bedford MK44 1LQ (GB)

(54) **Fabric softening composition.**

(57) A fabric softening composition comprising a water insoluble cationic fabric softening agent and a nonionic stabilising agent wherein the water insoluble cationic fabric softening agent is a biodegradable quaternary ammonium material with at least one ester link and the nonionic stabilising agent is selected from :

i. a linear C_8 to C_{22} alcohol alkoxylated with 10 to 20 moles of alkylene oxide, or

ii. a C_{10} to C_{20} alcohol or mixtures thereof.

The invention also provides a process for making the liquid fabric softening composition comprising the steps of

i. mixing and heating the cationic fabric softening agent and nonionic stabilising agent to form a melt and

ii. dispersing the melt in water.

EP 0 507 478 A1

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 and a nonionic stabilising agent suitable as rinse-added fabric softener compositions.

Rinse added fabric softener compositions are well known. Typically such compositions contain a water insoluble quaternary ammonium fabric softening agent dispersed in water at a level of softening agent up to 7% by weight in which case the compositions are considered dilute, or at levels from 7% to 50% in which case the compositions are considered concentrates. In addition to softening, fabric softening compositions desirably have other benefits. One is the ability to confer soil release properties to fabrics, particularly those woven from polyester fibres.

One of the problems associated with fabric softening compositions is the physical instability of such compositions when stored. This problem is accentuated by having a concentrated composition and by storage at low temperatures.

Concentrates and storage stability at low temperatures are however desired by the consumer. Physical instability manifests as a thickening on storage of the composition to a level where the composition is no longer pourable and can even lead to the formation of an irreversible gel. The thickening is very undesirable since the composition can no longer be conveniently used.

In the past physical stability of rinse added fabric softener compositions has been improved by the addition of viscosity control agents or anti-gelling agents. For example in EP 13780 (Procter and Gamble) viscosity control agents are added to certain concentrated compositions. The agents may include C_{10}-C_{18} fatty alcohols. More recently in. EP 280550 (Unilever) it has been proposed to improve the physical stability of dilute compositions comprising biodegradable, ester-linked quaternary ammonium compounds and fatty acid by the addition of nonionic surfactants.

With concentrated compositions comprising biodegradable ester-linked quaternary ammonium compounds the problem of physical instability is more acute than with traditional quaternary ammonium compounds.

In EP 0 040 562 (Lesieur Cotelle) a nonionic emulsifier/stabiliser is added to a concentrate comprising an ester-linked quaternary ammonium compound to form a viscous gel. The stabiliser is a C_{12} to C_{14} alcohol ethoxylated with 9 molecules of ethylene oxide. The degree of branching of the alcohol is not, however, mentioned.

Certain nonionic stabilising agents not only stabilise concentrated compositions comprising biodegradable quaternary ammonium compounds but are also environmentally friendly, in that they show acceptable biodegradability and are not substantially toxic in aquatic systems.

Soil release properties are generally imparted to fabrics by the use of separate soil-release agents, usually a high molecular weight polymer, in a detergent composition or separate treatment. For example in EP 0 398 133A (Procter & Gamble) there is disclosed a cationic polymeric soil release agent for use in a fabric conditioning composition.

A disadvantage of such compositions is that the soil release agent increases the number of components in the formulation, increasing cost and making the product less environmentally acceptable.

We have now found that fabric softening compositions comprising biodegradable ester-linked quaternary ammonium compounds may confer improved soil release properties to fabrics.

Thus, according to one aspect of the invention there is provided a fabric softening composition comprising a water insoluble cationic fabric softening agent and a nonionic stabilising agent characterised in that the water insoluble cationic fabric softening agent is a biodegradable quaternary ammonium material with at least one ester link and the nonionic stabilising agent is

i. a linear C_8 to C_{22} alcohol alkoxylated with 10 to 20 moles of alkylene oxide

or

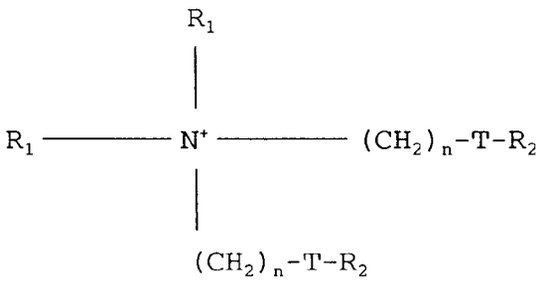
ii. a C_{10} to C_{20} alcohol or mixtures thereof.

Advantageously the nonionic stabilising agent is a linear C_8 to C_{22} alcohol alkoxylated with 10 to 20 moles of alkylene oxide

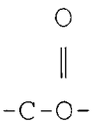
Preferably the compositions of the invention are liquids comprising an aqueous base.

Advantageously the fabric softening composition comprises a water insoluble cationic which is a compound having two C_{12-28} alkyl or alkenyl groups connected to the N atom via one or more ester links.

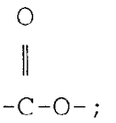
A preferred type of ester-linked quaternary ammonium material for use in the compositions according to the invention can be represented by the formula:



wherein each R_1 group is independently selected from C_{1-4} alkyl, alkenyl or hydroxyalkyl groups; and wherein each R_2 group is independently selected from C_{8-28} alkyl or alkenyl groups;
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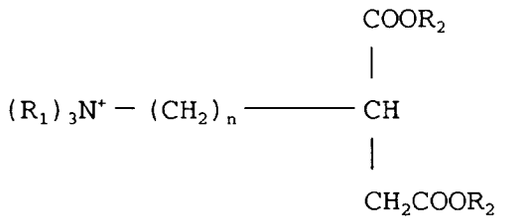


or



and
n is an integer from 0-5.

A second preferred type of quaternary ammonium material can be represented by the formula:



wherein R_1, n and R_2 are as defined above.

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 amounts of the corresponding monoester as described in US 4 137 180 for example 1-tallowoxy, 2-hydroxytrimethyl ammonium propane chloride.

Preferably the level of ester linked quaternary ammonium compounds is at least 1% by weight of the composition, more 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 preferably 3% to 50%, most preferably 8% to 50%.

Suitable nonionic stabilisers which can be used include the condensation products of C_8 - C_{22} primary linear alcohols with 10 to 20 moles of ethylene oxide. The term linear alcohol means a primary alcohol attached directly to a hydrocarbon backbone structure. Use of less than 10 moles of ethylene oxide, especially when the alkyl chain is in the tallow range, leads to unacceptably high aquatic toxicity. Since the aquatic toxicity is related to both the number of moles of ethylene oxide and the length of the alkyl chain we have found that the HLB value can be used as an indication of likely aquatic toxicity. An HLB of greater than about 12 gives rise to an accept-

able acute aquatic toxicity value of >1mg/1;EC_{50} 48 hours for daphnia and algae and EC_{50} 96 hours for fish. The selection of linear alcohols and the use of 20 moles or less of ethylene oxide gives acceptable biodegradability to the nonionic stabiliser. However the use of nonionic stabilisers with more than 20 ethylene oxide units will also provide the stability benefit .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, 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 20, more preferably 12 and 20.

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 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.

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 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 compositions of the invention preferably have a pH of more than 2.0, more preferably less than 5.

The composition can also contain one or more optional ingredients, selected from non-aqueous solvents, pH buffering agents, perfumes, perfume carriers, fluorescers, colourants, 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.

The invention will now be illustrated by the following non-limiting examples. In the examples all percentages are expressed by weight.

Example 1

Liquid fabric softening compositions were made as follows.

The cationic fabric softening agent, fatty acid and nonionic stabilising agent where appropriate were premixed and heated together to form a clear melt. The molten mixture thus formed was added over a period of at least one minute, to water at 70°C to 80°C with constant stirring to form a dispersion.

The viscosity of the compositions was measured by Haake rotoviscometer following 21 days storage at ambient temperature or at 5°C.

Composition	A	B	C	D	E
Arquad 2HT[1]	12.8	-	-	-	-
HT TMPAC[2]	-	16	16	16	16
Fatty acid[3]	3.2	2.7	2.7	2.7	2.7
Tallow 11EO[4]	-	3	-	-	-
Coco 10EO[5]	-	-	2	-	-
Tallow 20EO[6]	-	-	-	3	-
Water + minors	----------balance----------				
Viscosity at $110s^{-1}$					
Ambient mPas	159	77	34	43	90
5°C mPas	155	66	38	47	gel

Composition	F	G
HT TMPAC[2]	11.6	12.6
Fatty acid[3]	1.9	2.1
Alcohol[7]	1.5	1.5
Water + minors	--balance---	
Viscosity at $110s^{-1}$		
Ambient mPas	53	50
5°C mPas	50	50

Notes

Formulation A corresponds to a commercially available fabric softening composition currently sold in the UK by Lever under the trade mark COMFORT.

1. Arquad 2HT is dihardened tallow dimethyl ammonium chloride ex Akzo Chemie.
2. HT TMAPC is 1,2 dihardened tallowyloxy-3-trimethyl-ammonio propane chloride ex Hoescht.
3. Pristerine 4916 a hardened tallow fatty acid ex Unichema.
4. tallow alcohol ethoxylated with 11 moles of ethylene oxide of HLB 13.
5. coco alcohol ethoxylated with 10 moles of ethylene oxide of HLB 14.
6. tallow alcohol ethoxylated with 20 moles of ethylene oxide of HLB 20.
7. Laurex CS a tallow alcohol comprising 65-80% C_{18} and 20-23% C_{16} ex Albright and Wilson.

The results show that while known fabric softening compositions comprising conventional quaternary ammonium compounds do not show physical instability on short term storage at 5°C (composition A) a problem is experienced with compositions comprising ester-linked quaternaries and fatty acid (composition E). The results also show that addition of selected nonionic stabilising agents counteract destabilisation to give stable concentrated compositions.

Example 2

Liquid fabric softening compositions as given below were made as described in Example 1. The soil release properties imparted to polyester test pieces by treatment with the compositions was assessed by measuring the change in reflectance following staining and a subsequent wash in a proprietary detergent composition. The pieces were first rinsed for 5 minutes in 1 litre of 14°FH water containing 0.67 ml of either composition. The pieces were then line dried and stained with 100 micro litres of olive oil containing 0.06% sudan red dye. The stain was allowed to spread for a minimum of two days following which the reflectance of the stained piece (R_1) was measured using an ICS micromatch. The pieces were then washed, rinsed and line dried using 5g/l New System Persil Automatic ex Lever in 14°FH water for a 15 minute wash cycle. The reflectance of the pretreated, washed piece (R_2) was measured and the percentage detergency calculated according to the following equation:

$$\% \text{ Detergency} = \frac{Ks_1 - Ks_2}{Ks_1} \times 100$$

$$\text{where } Ks_1 = \frac{(1 - R_1)^2}{2R_1} \text{ and } Ks_2 = \frac{(1 - R_2)^2}{2R_2}$$

The higher the percentage detergency, the greater the soil release benefit.

Composition	% by weight	
	A	B
Arquad 2HT[1]	-	12.8
HT TMAPC[2]	11.6	-
Fatty acid[3]	1.9	3.2
Tallow 11EO[4]	2.5	-
Water and minors to balance		
% Detergency	30	21

Notes

Formulation B corresponds to a commercially available fabric softening composition, currently sold in the UK by Lever under the trade mark COMFORT. 1, 2, 3 and 4 are as in Example 1.

These results show that known compositions comprising conventional quaternary ammonium compositions (Composition B) show a smaller soil release benefit than compositions according to the invention (Composition A).

Example 3

Preferred compositions according to the invention are as follows:

Composition	% by weight		
	A	B	C
HTTMAPC[2]	11.6	11.6	11.6
Fatty acid[3]	1.9	1.9	1.9
Tallow 11EO[4]	-	2.5	-
Tallow Alcohol[7]	1.5	-	-
Tallow 15EO[8]	-	-	1.5
Isopropyl alcohol	1.6	1.6	1.6
Glycerol	1.6	1.6	1.6
Perfume, Dye + minors	0.8	0.8	0.8
Water to balance			

Notes

2, 3, 4 and 7 are as in Example 1

8. is tallow alcohol ethoxylated with 15 moles of ethylene oxide.

Claims

1. A fabric softening composition comprising a water insoluble cationic fabric softening agent and a nonionic stabilising agent characterised in that the water insoluble cationic fabric softening agent is a biodegradable quaternary ammonium material with at least one ester link and the nonionic stabilising agent is
   i. a linear C_8 to C_{22} alcohol alkoxylated with 10 to 20 moles of alkylene oxide, or
   ii. a C_{10} to C_{20} alcohol or mixtures thereof.

2. A composition as claimed in claim 1 wherein the nonionic stabilising agent is a linear C_8 to C_{22} alcohol alkoxylated with 10 to 20 moles of alkylene oxide.

3. A composition as claimed in claim 1 or claim 2 characterised in that the composition comprises at least 1% by weight of the quaternary ammonium material.

4. A composition as claimed in any preceding claim characterised in that the composition comprises from 0.1 to 10% by weight of the nonionic stabilising agent.

5. A composition as claimed in any preceding claim characterised in that the composition also comprises more than 0.1% by weight of a fatty acid material.

6. A composition as claimed in any preceding claim characterised in that the composition comprises from 3% to 50% by weight of the quaternary ammonium material, from 0.5% to 5% by weight of the nonionic stabilising agent and from 0.5 to 20% by weight of fatty acid material.

7. A composition as claimed in any preceding claim characterised in that the nonionic stabilising agent has an HLB of between 10 and 20.

8. A composition as claimed in claim 7 wherein the nonionic stabilising agent has an HLB of between 12 and 20.

9. A fabric softening composition as claimed in any preceding claim wherein the water insoluble cationic com-

prises a compound having two C_{12-28} alkyl or alkenyl groups connected to the N atom via one or more ester links.

10. Process for making a liquid fabric softening composition as claimed in any of claims 1 to 9 comprising the steps of

i. mixing and heating the cationic fabric softening agent and nonionic stabilising agent to form a melt and

ii. dispersing the melt in water.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 92 30 2452

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	EP-A-0 293 953 (PROCTER & GAMBLE) * Page 4, line 57 - page 5, line 2; example 6; claim 1 *	1-4,7-10	C 11 D 1/835 C 11 D 1/62
X	EP-A-0 309 052 (PROCTER & GAMBLE) * Page 8, line 34 - page 9, line 4; page 13, line 19; example 1; claims *	1-5,9,10	
X	US-A-4 789 491 (CHANG et al.) * Column 6, lines 14-17; examples; claims *	1,3,9	
X	EP-A-0 346 634 (HENKEL) * Page 4, lines 4-6; claims *	1-3,5	
X	EP-A-0 331 237 (UNILEVER) * Page 3, line 41 - page 4, line 45; claims *	1,3	
A	FR-A-2 257 728 (PROCTER & GAMBLE) * Claims *	1	

TECHNICAL FIELDS SEARCHED (Int. Cl.5)

C 11 D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	26-06-1992	GOLLER P.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
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T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

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