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(54)**Fabric softening composition**

(57)A fabric softening composition with good storage stability and excellent perfume stability is disclosed. The composition contains a cationic softening agent and a perfume/carrier substance with a slip point below 45°C.

Description

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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 perfume. The compositions are stable on storage and exhibit excellent perfume delivery when removed from a washing machine.

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 such as delivering perfume onto fabrics.

The fabric conditioning market is moving towards more concentrated compositions. One of the problems associated with such fabric softening compositions is that on storage they are physically unstable. This problem is accentuated by the presence of a perfume; the greater the level of perfume the worse the stability problem becomes.

Physical instability manifests itself as a irreversible thickening on storage of the composition to a level where the composition is no longer pourable and can even lead to the formation of a gel. The thickening is very undesirable since the composition can no longer conveniently be 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 13 780 (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 280 550 (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. EP 507 478 (Unilever) discloses a physically stable fabric softening composition comprising a water insoluble, biodegradable, ester-linked quaternary ammonium compounds and a nonionic stabilising agent.

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

CA 1 111 616 (Procter and Gamble) discloses a fabric softening composition comprising 0.1 to 10 wt% of a cationic fabric softener and 0.1 wt% to 20 wt % of particles. The particles are comprised of a carrier substance and a perfume and have a melting point of from 38°C to 150°C, preferably from 65°C to 100°C.

We have now found that fabric softening compositions comprising quaternary ammonium compounds may exhibit excellent perfume substantivity and good storage stability.

Thus, according to one aspect of the invention there is provided a liquid fabric softening composition a fabric softening composition comprising:

- i) a quaternary ammonium fabric softening agent,
- ii) from 0.1 wt% to 10 wt% of a perfume/carrier substance mix having a slip point below 45°C.

The slip point of the perfume/carrier substance mix is measured as defined by British Standard BS 684 section 1.3 1991 ISO 6321:1991 (UK).

A further aspect of the invention is provided by a process for preparing a fabric softening composition as defined above, which comprises of the following steps:

- i) forming an aqueous dispersion of the quaternary ammonium fabric softening agent;
- ii) adding the perfume/carrier substance mix to the resulting dispersion.

One advantage of such a process is that it is simple. A further advantage afforded by the present invention is that the degree of fabric softening may be varied to suit different customer requirements in different markets, while maintaining high perfume delivery.

The invention also allows perfume to be incorporated without excessive perfume intensity from the undiluted product.

To give excellent perfume delivery when laundry is removed from the washing machine it is advantageous if the perfume carrier substance mix has a slip point below 37°C; preferably from 0 to 37°C. More preferably it has a slip point between 20°C and 37°C.

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

It is preferred if the carrier substance is an organic material. It is further preferred if the carrier substance is a fatty ester of a mono or polyhydric alcohol having from 1 to about 24 carbon atoms in the hydrocarbon chain with the proviso that the total number of carbon atoms in the carrier substance is equal to or greater than 16 and that at least one of the hydrocarbons radicals in the ester has 12 or more carbon atoms. Such fatty esters are preferably odourless.

It is preferred if the carrier substance in the perfume/carrier substance mix is a fatty acid glyceride or mixtures of

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fatty acid glycerides. Especially preferred carrier substances are triglycerides, most preferred are palm oil and tallow oil.

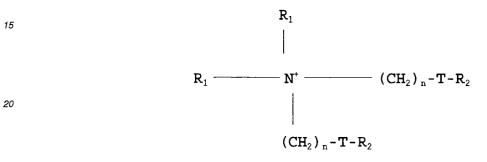
The perfume/carrier substance mix is preferably present at a level of from 1 to 10% of the composition, more preferably 3 wt% to 7 wt% of the total composition.

For good perfume delivery it is advantageous if the ratio of perfume to carrier substance is from 1:50 to 1:1, more preferably 1:20 to 1:2, most preferably 1:7 to 1:2.

To achieve good perfume delivery from the product it is favourable if the ratio of perfume/carrier substance mix to quaternary ammonium fabric softening agent is in the ratio of 5:1 to 1:10, more preferably 2:1 to 1:10.

It is preferable if the quaternary ammonium compound is biodegradable and has at least one ester link. Advantageously the fabric softening composition comprises a water insoluble cationic 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 fabric softening agent 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, or hydroxyalkyl or C_{2-4} alkyl groups; and wherein each R_2 group is independently selected from C_{8-28} alkyl or alkenyl groups;

T is

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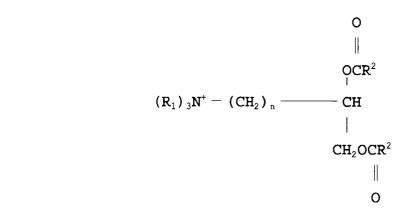
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0 0 || || -C-O- or -C-O

and

n is an integer from 0-5.

A second preferred type of quaternary ammonium fabric softening agent can be represented by the formula:



wherein R₁, n and R₂ 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

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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 7% by weight of the composition; especially interesting are concentrated compositions which comprise between 10 and 30 % by weight of ester-linked quaternary ammonium compound.

Compositions of the invention may also contain nonionic stabilisers. Suitable nonionic stabilisers which can be used include the condensation products of C_8 - C_{22} primary linear alcohols with 10 to 25 moles of ethylene oxide. 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. In particular the following nonionic stabilisers are preferred: Genapol T-110, Genapol T-150, Genapol T-200, Genapol C-200 all ex Hoechst AG, Lutensol AT18 ex BASF, Genapol C-100 and Genapol C-150 ex Hoechst, or fatty alcohols for example Laurex CS, ex Albright and Wilson or Adol 340 ex Sherex (all Trade Marks). Preferably the nonionic stabilizer has an HLB value of from 10 to 20, more preferably 12 to 20. Preferably, the level of nonionic stabiliser is within the range of 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 composition can also contain fatty acids, for example C_8 - C_{24} alkyl or alkenyl monocarboxylic acids, or polymeric carboxylic acids. Preferably saturated fatty acids are used, in particular, hardened tallow C_{16} - C_{18} fatty acids.

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 1.5, 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, 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.

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

Comparative Examples are designated by letters, while Examples of the invention are designated by numbers.

Examples 1, 2, 3, 4, A, B, C and D

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Compositions were prepared by the following method: antifoam, preservative and dyestuff were dissolved in warm (40°C) water followed by addition of electrolyte (citric or CaCl₂)to form an aqueous solution. The biodegradable cationic fabric softening agent (HT TMAPC = 1-trimethyl ammonium-2,3 dihardened tallowoyloxy propane chloride with some tallow fatty acid impurities (6:1 ratio) ex Hoechst) was melted (75-80°C) and mixed with the nonionic (coco ethoxylate 20EO:coco 20). This premix was added to the aqueous solution over 5 min with stirring. The resulting mixture was recirculated through a Janke and Kunkel mill set at max shear (10,000 rpm). This recirculation/shearing continued for 30 mins after premix addition. Perfume was stirred directly into the mix or a premixed with palm/tallow oil at a temperature of 40-45°C and then added by milling into the product over a 10 min period at 8000 rpm.

(The palm/tallow raw materials are refined, bleached and deodorised).

TABLE 1

Formulations	EXAMPLES			
	Α	1	В	2
Citric Acid	3.5	3.5	3.5	3.5
HT TMAPC	20	20	20	20
Nonionic (Coco 20)	1.0	1.0	3.0	3.0
Antifoam	0.06	0.06	0.06	0.06
Preservative	0.02	0.02	0.02	0.02
Perfume	1.0	1.0	1.0	1.0
Palm Oil	-	3.0	-	-
Tallow Oil	-	-	-	3.0
Demin Water	to 100	to 100	to 100	to 100

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TABLE 2

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D

4

С

Example

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CaCl₂ 0.20 0.20 0.20 0.20 HT TMAPC 20 20 20 20 Nonionic (Coco 20) 3.0 3.0 3.0 3.0 Antifoam 0.06 0.06 0.06 0.06 Preservative 0.02 0.02 0.02 0.02 Perfume 1.0 1.0 1.0 1.0 Palm Oil -3.0 --Tallow Oil 5.0 Demin Water to 100 to 100 to 100 to 100

Viscosity measurements were taken at 25° and at 106 sec⁻¹, shear rate using a Haake VT501 rotoviscometer. Examples A and B went solid within 3 days at 37°C storage. Examples 1 and 2 showed viscosities as follows:

TABLE 3

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Time @ 37°C	Viscosity		
	1	2	
Initial	69 cP	118 cP	
10 days	-	122 cP	
12 days	65 cP	-	
22 days	-	125 cP	
28 days	93 cP	-	
29 days	-	121 cP	
38 days	-	118 cP	
66 days	-	129 cP	

Examples C and D went solid within 3hrs at ambient temperature. Examples 3 and 4 showed viscosities as follows:

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TABLE 4

Time @ 37°C	Visc		
	3	4	
Initial	64 cP	38 cP	
7 days	-	34 cP	
10 days	68 cP	-	
19 days	-	33 cP	
21 days	82 cP	-	
43 days	-	36 cP	
56 days	87 cP	-	
58 days	-	39 cP	
71 days	99 cP	-	

Examples 5,6.7.8.E,F,G and H

To demonstrate that this invention works with a variety of perfumes, 300g of an unperfumed base (at 35 cP) of composition 4 was prepared with either perfume alone or perfume/palm oil mix by milling into the base using a Silverson mixer over a 5 min period. The samples were left at ambient temperature.

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TABLE 5

Examples	Perfume	Palm Oil	Time to go Solid
Е	Perfume A	-	10 mins
5	Perfume A	3%	Liquid after 10 days
F	Perfume B	-	2-3 days
6	Perfume B	3%	Liquid after 10 days
G	Perfume C	-	10 mins
7	Perfume C	3%	Liquid after 10 days
Н	Perfume D	-	1-2 days
8	Perfume D	3%	Liquid after 10 days

Examples 9,10 and I

Examples were prepared by dispersing the cationic fabric softening agent in water, followed by the addition of the perfume or perfume/carrier substance mix.

The perfume substantivity was tested by adding 1g of the resulting composition to 1 litre of water at ambient temperature and agitating for 2 minutes to disperse. Four 19 x 19 squares of terry towelling were than immersed in the dispersion and left for 5 minutes. The terry towelling was then removed from the dispersion, wrung dry and line dried overnight.

The terry towelling samples were evaluated by a panel of people using a random design to measure perfume strength.

Each treatment was evaluated six times. The data was analysed by analysis of variance.

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TABLE 6

Example	9	10	I
HT TMAPC	14.5	14.5	14.5
Perfume Softline 2000	0.9	0.9	0.9
Palm oil	5.0	2.7	-
Water	to 100		

The perfume release on the terry towelling was as illustrated in the table below:

TABLE 7

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	Example		
	9	10	I
Perfume Impact	1.5	1.7	0.5

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A score of 0 indicates a no perfume impact.

A score of 5 indicates a very strong perfume impact.

Claims

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- 1. A liquid fabric softening composition comprising:
 - i) a quaternary ammonium fabric softening agent and;
 - ii) from 0.1 wt% to 10 wt% of a perfume/carrier substance mix having a slip point below 45°C.

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- 2. A liquid fabric softening composition according to claim 1 in which the perfume/carrier substance mix has a slip point below 37°C.
- 3. A liquid fabric softening composition according to claim 1 or claim 2 in which the carrier substance is an organic material.
 - 4. A liquid fabric softening composition according to any preceding claim in which the carrier substance is a fatty ester of a mono or polyhydric alcohol having from 1 to about 24 carbon atoms in the hydrocarbon chain with the proviso that the total number of carbon atoms in the carrier substance is equal to or greater than 16 and that at least one of the hydrocarbons radicals in the ester has 12 or more carbon atoms.
 - 5. A liquid fabric softening composition according to any preceding claim in which the carrier substance is odourless.
 - 6. A liquid fabric softening composition according to claim 1 in which the carrier substance is tallow oil.

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- 7. A liquid fabric softening composition according to any preceding claim in which the ratio of perfume/carrier substance mix to quaternary ammonium fabric softening agent is in the ratio of 5:1 to 1:10
- 8. A liquid fabric softening composition according to any preceding claim in which the quaternary ammonium fabric softening agent is a biodegradable quaternary ammonium material with at least one ester link.
 - 9. A liquid fabric softening composition according to any preceding claim which is in the form of an aqueous dispersion.