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(54) **Fabric softening composition**

Gewebeweichmacherzusammensetzung

Composition adoucissante pour textiles

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WO-A-91/13143 **WO-A-93/05137**
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Description

[0001] 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.

[0002] 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.

[0003] 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.

[0004] Physical instability manifests itself as an 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.

[0005] 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 C₁₀-C₁₈ 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.

[0006] With concentrated compositions comprising biodegradable ester-linked quaternary ammonium compounds the problem of physical instability is more acute than with traditional quaternary ammonium compounds.

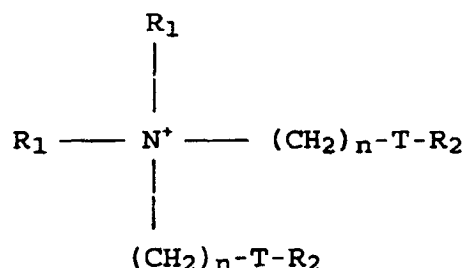
[0007] CA 1 111 616 (Procter and Gamble) discloses a fabric softening composition comprising 0.1 to 10 wt% of a cationic fabric softener and 0.1 wt% to 20 wt % of particles. The particles are comprised of a carrier substance and a perfume and have a melting point of from 38°C to 150°C, preferably from 65°C to 100°C.

[0008] We have now found that fabric softening compositions comprising quaternary ammonium compounds may exhibit excellent perfume substantivity and good storage stability.

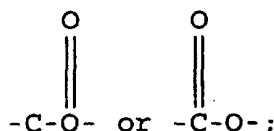
[0009] Thus, according to one aspect of the invention there is provided a liquid fabric softening composition a fabric softening composition comprising:

i) a quaternary ammonium fabric softening agent,

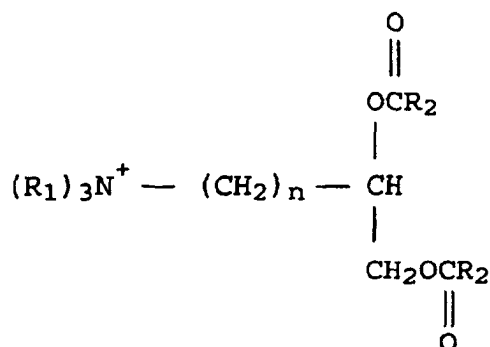
ii) from 0.1 wt% to 10 wt% of a perfume/carrier substance mix having a slip point below 37°C; in which the quaternary ammonium fabric softening agent is either



wherein each R₁ group is independently selected from C₁₋₄ alkyl, or hydroxyalkyl or C₂₋₄ alkenyl groups; and wherein each R₂ is independently selected from C₈₋₂₈ alkyl or alkenyl groups; T is



and n is an integer from 0-5; or



wherein R_1 , n and R_2 are as defined above; and further wherein the ratio of perfume to carrier substance is from 1:50 to 1:1.

[0010] 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).

[0011] 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.

[0012] 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.

[0013] The invention also allows perfume to be incorporated without excessive perfume intensity from the undiluted product.

[0014] To give excellent perfume delivery when laundry is removed from the washing machine, 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.

[0015] The compositions of the invention are liquids, more preferably comprising an aqueous base.

[0016] 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.

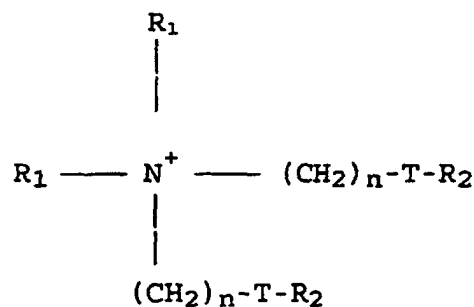
[0017] It is preferred if the carrier substance in the perfume/carrier substance mix is a fatty acid glyceride or mixtures of fatty acid glycerides. Especially preferred carrier substances are triglycerides, most preferred are palm oil and tallow oil.

[0018] 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.

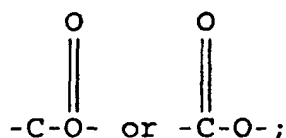
[0019] For good perfume delivery, the ratio of perfume to carrier substance is from 1:50 to 1:1, more preferably 1:20 to 1:2, most preferably 1:7 to 1:2.

[0020] 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.

[0021] 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:

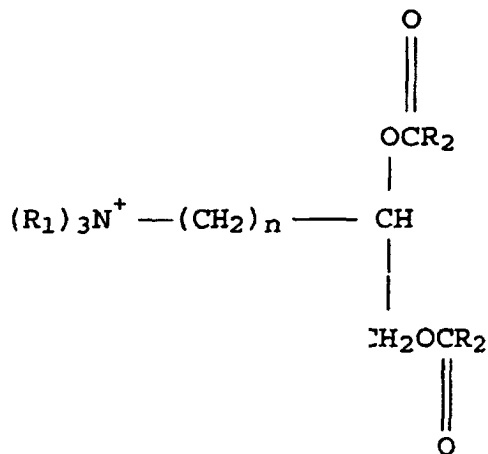


wherein each R_1 group is independently selected from C_{1-4} alkyl, or hydroxyalkyl or C_{2-4} alkyl groups; and wherein each R_2 group is independently selected from C_{8-28} alkyl or alkenyl groups; T is



and

n is an integer from 0-5; or by the formula:



wherein R_1 , n and R_2 are as defined above.

[0022] 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.

[0023] 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.

[0024] 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 stabiliser 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.

[0025] 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.

[0026] 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.

[0027] The compositions of the invention preferably have a pH of more than 1.5, more preferably less than 5.

[0028] The composition can also contain one or more optional ingredients, selected from non-aqueous solvents, pH buffering agents, 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.

[0029] The composition may also contain nonionic fabric softening agents.

[0030] The invention will now be illustrated by the following nonlimiting examples. In the examples all percentages are expressed by weight.

[0031] 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

[0032] 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 $CaCl_2$) to form an aqueous solution. The biodegradable cationic fabric softening agent (HT TMAPC = 1-trimethyl ammonium-2,3 dihardened tallowoxyloxy 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 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.

[0033] (The palm/tallow raw materials are refined, bleached and deodorised).

TABLE 1

Formulations	EXAMPLES			
	A	1	B	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

TABLE 2

Example	C	3	D	4
$CaCl_2$	0.20	0.20	0.20	0.20

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TABLE 2 (continued)

Example	C	3	D	4
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

[0034] Viscosity measurements were taken at 25°C and at 106 sec⁻¹, shear rate using a Haake VT501 rotoviscometer.

[0035] Examples A and B went solid within 3 days at 37°C storage. Examples 1 and 2 showed viscosities as follows:

TABLE 3

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

[0036] Examples C and D went solid within 3hrs at ambient temperature.

Examples 3 and 4 showed viscosities as follows:

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	-

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Examples 5,6,7,8,E,F,G and H

[0037] 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.

TABLE 5

Examples	Perfume	Palm Oil	Time to go Solid
E	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
H	Perfume D	-	1-2 days
8	Perfume D	3%	Liquid after 10 days

Examples 9,10 and I

[0038] Examples were prepared by dispersing the cationic fabric softening agent in water, followed by the addition of the perfume or perfume/carrier substance mix.

[0039] 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 then 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.

[0040] Each treatment was evaluated six times. The data was analysed by analysis of variance.

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 -----		

[0041] The perfume release on the terry towelling was as illustrated in the table below:

TABLE 7

	Example		
	9	10	I
Perfume Impact	1.5	1.7	0.5

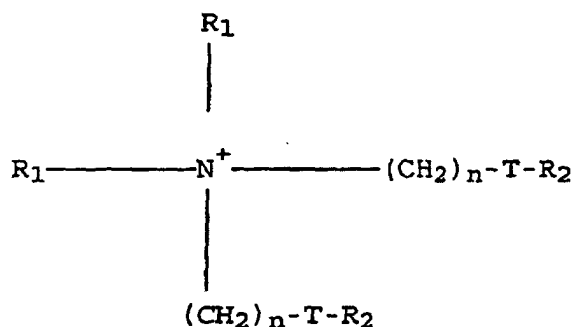
[0042] A score of 0 indicates a no perfume impact.

[0043] A score of 5 indicates a very strong perfume impact.

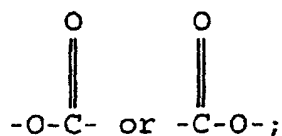
Claims

1. A liquid fabric softening composition comprising:

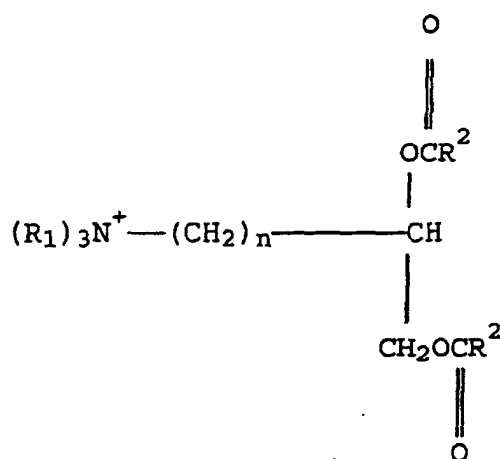
- i) a quaternary ammonium fabric softening agent and;
 ii) from 0.1 wt% to 10 wt% of a perfume/carrier substance mix having a slip point below 37°C; in which the quaternary ammonium fabric softening agent is either



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



and
 n is an integer from 0-5; or



wherein R_1 , n and R_2 are as defined above and the ratio of perfume to carrier substance is from 1:50 to 1:1.

2. A liquid fabric softening composition according to claim 1 in which the carrier substance is an organic material.

3. 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.

4. A liquid fabric softening composition according to claim 1 in which the carrier substance is tallow oil.

5. 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.

6. A liquid fabric softening composition according to any preceding claim which is in the form of an aqueous dispersion.

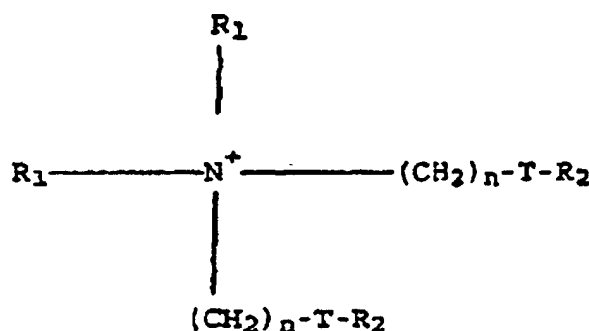
7. A process for preparing a liquid fabric softening composition as defined in any one of the preceding claims comprising of the following steps:

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

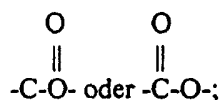
Patentansprüche

1. Flüssige Gewebeweichmacherzusammensetzung, umfassend:

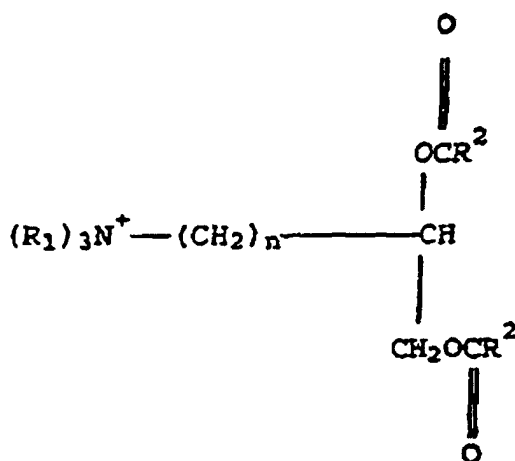
- i) einen quaternären Ammoniumgewebeweichmacher; und
- ii) 0,1 Gew.-% bis 10 Gew.-% eines Duftstoff-/Trägersubstanzgemisches mit einem Fließschmelzpunkt unter 37 °C; worin ist der quaternäre Ammoniumgewebeweichmacher entweder



ist, wobei jede R_1 -Gruppe unabhängig ausgewählt ist aus C_{1-4} Alkyl- oder Hydroxyalkyl- oder C_{2-4} Alkenylgruppen; und wobei jede R_2 -Gruppe unabhängig ausgewählt ist aus C_{8-28} Alkyl- oder Alkenylgruppen; T ist



und
n ist eine ganze Zahl von 0-5; oder



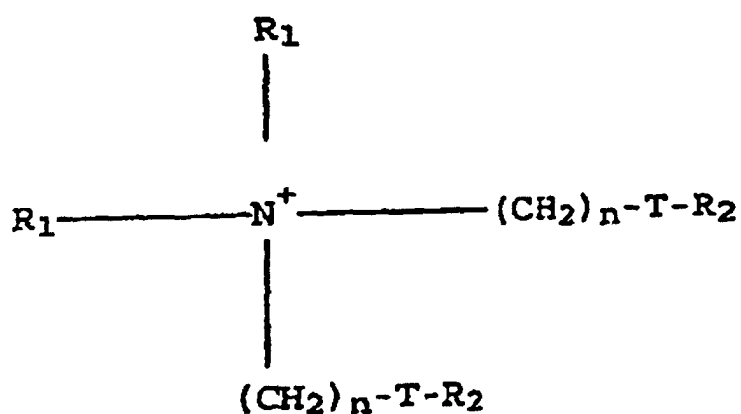
wobei R_1 , n und R_2 wie oben definiert sind und das Verhältnis von Duftstoff zu Trägersubstanz 1:50 bis 1:1 ist.

2. Flüssige Gewebeweichmacherzusammensetzung gemäß Anspruch 1, bei der die Trägersubstanz ein organisches Material ist.
3. Flüssige Gewebeweichmacherzusammensetzung gemäß einem der vorstehenden Ansprüche, bei der die Trägersubstanz ein Fettester eines ein- oder mehrwertigen Alkohols mit 1 bis etwa 24 Kohlenstoffatomen in der Kohlenwasserstoffkette ist, mit der Bedingung, dass die Gesamtzahl an Kohlenstoffatomen in der Trägersubstanz gleich oder größer als 16 ist und dass mindestens einer der Kohlenwasserstoffreste im Ester 12 oder mehr Kohlenstoffatome aufweist.
4. Flüssige Gewebeweichmacherzusammensetzung gemäß Anspruch 1, bei der die Trägersubstanz Talgöl ist.
5. Flüssige Gewebeweichmacherzusammensetzung gemäß einem der vorstehenden Ansprüche, bei der das Verhältnis von Duftstoff-/Trägersubstanzgemisch zu quaternärem Ammoniumgewebeweichmacher 5:1 bis 1:10 ist.
6. Flüssige Gewebeweichmacherzusammensetzung gemäß einem der vorstehenden Ansprüche, die in der Form einer wässrigen Dispersion vorliegt.
7. Verfahren zur Herstellung einer flüssigen Gewebeweichmacherzusammensetzung wie in einem der vorstehenden Ansprüche definiert, das folgende Schritte umfasst:
 - (i) Bildung einer wässrigen Dispersion des quaternären Ammoniumgewebeweichmachers;
 - (ii) Hinzufügen des Duftstoff-/Trägersubstanzgemisches zu der resultierenden Dispersion.

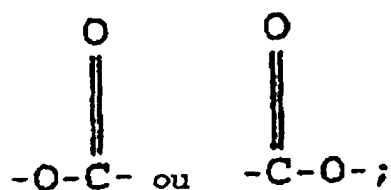
Revendications

1. Composition adoucissante liquide pour les textiles comprenant :

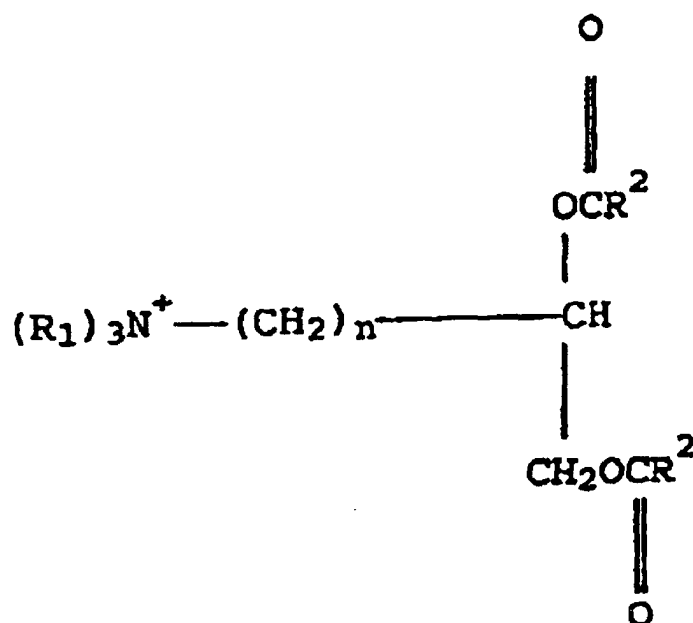
- i) un agent d'adoucissement des textiles ammonium quaternaire et ;
- ii) de 0,1 % en poids à 10 % en poids d'un mélange parfum/substance véhicule ayant un point de glissement en dessous de 37°C ; dans laquelle l'agent d'adoucissement des textiles ammonium quaternaire est soit



dans laquelle chaque groupe R_1 est indépendamment choisi parmi les groupes alkyle en C_{1-4} , ou hydroxyalkyle ou les groupes alkyle en C_{2-4} ; et dans laquelle chaque groupe R_2 est indépendamment choisi parmi les groupes alkyle ou alcényle en C_{8-28} ; T est



et
 n est un entier de 0 à 5; ou



dans laquelle R_1 , n et R_2 sont comme définis ci-dessus et le rapport parfum sur substance véhicule est de 1:50 à 1:1.

2. Composition adoucissante liquide pour les textiles selon la revendication 1, dans laquelle la substance véhicule est un matériau organique.
3. Composition adoucissante liquide pour les textiles selon l'une quelconque des revendications précédentes, dans laquelle la substance véhicule est un ester gras d'un alcool mono ou polyhydrique ayant de 1 à environ 24 atomes de carbone dans la chaîne hydrocarbonée à condition que le nombre total d'atomes de carbone dans la substance véhicule soit égal ou supérieur à 16 et qu'au moins un des radicaux hydrocarbonés dans l'ester ait 12 atomes de carbone ou plus.
4. Composition adoucissante liquide pour les textiles selon la revendication 1, dans laquelle la substance véhicule est de l'huile de suif.
5. Composition adoucissante liquide pour les textiles selon l'une quelconque des revendications précédentes, dans laquelle le rapport du mélange parfum / substance véhicule à l'agent d'adoucissement ammonium quaternaire est compris dans le rapport allant de 5:1 à 1:10.
6. Composition adoucissante liquide pour les textiles selon l'une quelconque des revendications précédentes qui est sous la forme d'une dispersion aqueuse.
7. Procédé de préparation d'une composition adoucissante liquide pour les textiles selon l'une quelconque des revendications précédentes qui comprend les stades suivants :
 - (i) formation d'une dispersion aqueuse de l'agent d'adoucissement des textiles ammonium quaternaire ;
 - (ii) addition du mélange parfum/substance véhicule à la dispersion résultante.