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

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(7) Applicant: UNILEVER NV
Burgemeester s'Jacobplein 1 P.O. Box 760
NL-3000 DK Rotterdam (NL)

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(2) Inventor: Blackmore, Eunice Sheila 61 Kentmere Drive Pensby Wirral L61 5XW (GB)

> Topping, Brian William 4 Chalfield Close Great Sutton South Wirral, L66 4US (GB)

(74) Representative: van Gent, Jan Paulus (NL) et al Unilever N.V., Patent Division Postbus 137 NL-3130 AC Vlaardingen (NL)

54 Fabric-softening composition.

 $\bigcirc$  A liquid fabric softening composition containing an aqueous base, not more than 8% of a cationic fabric softening agent, at least 0.2% of a C<sub>8</sub>-C<sub>24</sub> fatty acid and a nonionic surfactant. The mole ratio of the cationic fabric softening agent to the nonionic surfactant is within the range from 40:1 to 1:1. The nonionic surfactant is added to give a composition which is stable to freeze thaw cycling.

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### **Description**

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## FABRIC SOFTENING COMPOSITION

The present invention relates to a fabric softening composition, in particular a fabric softening composition containing a water-insoluble cationic fabric softening agent, a fatty acid and other nonionic surfactants.

One of the problems associated with fabric softening compositions is the physical instability of such compositions when stored. This problem is accentuated if storage occurs at various cycling temperatures including those below the freezing point, since irreversible gels can be formed.

It has been proposed (European Patent Specification No 21476) to add a protonated di-polyethoxy monoalkyl amine, a lower alcohol and, optionally, a nonionic fabric conditioning agent to control the physical stability of a composition containing a quaternary ammonium fabric softener, the alcohol being additional to that present in the softener raw material. The disadvantage with the compositions disclosed is that the physical stability is only achieved if both the amine and alcohol are added to the composition.

We have now found that the addition of a nonionic surfactant to a composition containing a cationic fabric softening agent and a fatty acid gives a composition which is stable after one and multiple freeze thaw cycles without the necessity of adding additional alcohol.

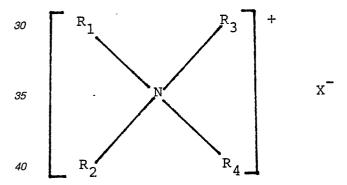
Thus, according to the invention there is provided a liquid fabric softening composition comprising (i) an aqueous base;

- (ii) not more than 8% by weight of a water-insoluble cationic fabric softening agent;
- (iii) at least 0.2% by weight of a C8-C24 fatty acid; and
- (iv) a nonionic surfactant,

wherein the mole ratio of the cationic fabric softening agent to the nonionic surfactant is within the range from 40:1 to about 1:1.

The water-insoluble cationic fabric softening agent can be any fabric-substantive cationic compound which has a solubility in water at pH 2.5 and  $20^{\circ}$ C of less than 10 g/l. Highly preferred materials are quaternary ammonium salts having two  $C_{12}$ - $C_{24}$  alkyl or alkenyl chains, optionally substituted or interrupted by functional groups such as -OH, -O-, -CONH-, -COO-, etc.

Well known species of substantially water-insoluble quaternary ammonium compounds have the formula



wherein  $R_1$  and  $R_2$  represent hydrocarbyl groups having from about 12 to about 24 carbon atoms;  $R_3$  and  $R_4$  represent hydrocarbyl groups containing from 1 to about 4 carbon atoms; and X is an anion, preferably selected from halide, methyl sulfate and ethyl sulfate radicals.

Representative examples of these quaternary softeners include ditallow dimethyl ammonium chloride; ditallow dimethyl ammonium methyl sulfate; dihexadecyl dimethyl ammonium chloride; di(hydrogenated tallow alkyl) dimethyl ammonium chloride; dioctadecyl dimethyl ammonium chloride; diecosyl dimethyl ammonium chloride; di(hydrogenated tallow) dimethyl ammonium methyl sulfate; dihexadecyl diethyl ammonium chloride; di(coconut alkyl) dimethyl ammonium chloride. Also suitable are dialkyl ethoxyl methyl ammonium sulphates based on soft or hard fatty acids. Ditallow dimethyl ammonium chloride, di(hydrogenated tallow alkyl) dimethyl ammonium chloride, di(coconut alkyl) dimethyl ammonium chloride and di(coconut alkyl) dimethyl ammonium methosulfate are preferred.

Another class of preferred water-insoluble cationic materials are the alkylimidazolinium salts believed to have the formula:

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wherein R<sub>6</sub> is an alkyl or hydroxyalkyl group containing from 1 to 4, preferably 1 or 2 carbon atoms, R<sub>7</sub> is an alkyl or alkenyl group containing from 8 to 25 carbon atoms, Re is an alkyl or alkenyl group containing from 8 to 25 carbon atoms, and R<sub>9</sub> is hydrogen or an alkyl containing from 1 to 4 carbon atoms and A<sup>-</sup> is an anion, preferably a halide, methosulfate or ethosulfate. Preferred imidazolinium salts include 1-methyl-1-(tallowylamido-) ethyl -2-tallowyi- 4,5-dihydro imidazolinlum methosulfate and 1-methyl-1- (palmitoylamido)ethyl -2-octadecyl-4,5- dihydro- imidazolinium chloride. Other useful imidazolinium materials are 2-heptadecyl-1-methyl-1- (2-stearylamido)- ethyl-imidazolinium chloride and 2-lauryl-1-hydroxyethyl- 1-oleyl-imidazolinium chloride. Also suitable herein are the imidazolinium fabric softening components of US Patent No 4 127 489, incorporated by reference. Mixtures of various cationic fabric softening agents can also be used.

The level of the cationic fabric softening agent in the composition is preferably more than 1% by weight, most preferably from 3% to 6% by weight.

Suitable fatty acids which can be used in the present invention are C8-C24 alkyl or alkenyl linear or branched chain monocarboxylic acids or polymers thereof. Preferably saturated fatty acids are used, in particular, hardened tallow C<sub>16</sub>-C<sub>18</sub> fatty acids. Mixtures of various fatty acids can also be used.

The level of the fatty acid in the composition is preferably less than 8% by weight, most preferably from 0.2% to 2.5% by weight.

The mole ratio of the cationic fabric softening agent to the fatty acid is at least 1:1, and preferably is within the range 4:1 to 9:1.

Suitable nonionic surfactants which can be used include in particular the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example aliphatic alcohols, acids, or alkyl phenois with alkylene oxides, especially ethylene oxide either alone or with propylene oxide. Specific nonionic surfactants are alkyl (C6-C22) phenols-ethylene oxide condensates, generally up to 25 EO, ie up to 25 units of ethylene oxide per molecule, the condensation products of aliphatic (C8-C22) primary or secondary linear or branched alcohols with ethylene oxide, generally up to 40 EO, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylenediamine. Other so-called nonionic surfactants include amine oxides, alkyl polyglycosides, ethoxylated castor oils, and sorbitan esters.

Preferably, the level of the nonionic surfactant is within the range from 0.1 to 4.5% by weight, most preferably from 0.15% to 3% by weight. The mole ratio of the cationic fabric softening agent to the nonionic surfactant 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 one or more optional ingredients selected from non-aqueous solvents such as C1-C4 alkanols and polyhydric alcohols (although the benefits of this invention can be obtained without the addition of these materials), pH buffering agents, such as weak acids eg phosphoric, benzoic or citric acids (the pH of the compositions are preferably less than 6.0), rewetting agents, viscosity modifiers, such as electrolytes and C<sub>9</sub>-C<sub>24</sub> fatty acids included at levels from about 20 to 6000 ppm, antigelling agents, perfumes, perfume carriers, fluorescers, colourants, hydrotropes, antifoaming agents, antiredeposition agents, enzymes, optical brightening agents, opacifiers, stabilisers such as guar gum and polyethylene glycol, anti-shrinking agents, anti-wrinkle agents, fabric crisping agents, anti-spotting agents, soil-release agents, germicides, fungicides, anti-oxidants, anti-corrosion agents, preservatives such as Bronopol (Trade Mark), a commercially available form of 2-bromo-2-nitropropane-1,3 diol, dyes, bleaches and bleach precursors, drape imparting agents, antistatic agents and ironing aids, such as silicones.

These optional ingredients, if added, are each present at levels up to 5% by weight of the composition. Suitable silicones for use in the compositions according to the invention include predominantly linear polydialkyl or alkylaryl siloxanes in which the alkyl groups contain one to five carbon atoms. The siloxanes can be amido or amino substituted. When the siloxane is amine substituted the amine group may be quaternised.

The compositions may also contain, in addition to the cationic fabric softening agent, other non-cationic fabric softening agents, such as nonionic fabric softening agents. Suitable nonionic fabric softening agents

include lanolin and derivatives thereof. Suitable materials are disclosed in European Patent Applications 88 520 (Unilever), the disclosure of which is incorporated herein by reference. Typically such materials are included at a level within the range of from 0.5% to 10% by weight of the composition.

In use, the fabric conditioning composition of the invention may be added to a large volume of water to form a liquor with which the fabrics to be treated are contacted. Generally, the total concentration of the cationic fabric softening agent, the fatty acid and the nonionic surfactant in this liquor will be between about 30 ppm and 500 ppm. The weight ratio of the fabrics to liquor will preferably be less than about 25:1, most preferably between about 10:1 and about 4:1.

The compositions of the invention may be prepared by a variety of methods. One suitable method is to form a molten mixture of the cationic fabric softening agent and the fatty acid, add this molten mixture to water with stirring to form a dispersion and thereafter add the nonionic surfactant and any optional ingredients. Another suitable method is to add the nonionic surfactant to the molten mixture before the dispersion is formed.

The invention will now be illustrated by the following non-limiting examples. In the examples all weights are expressed as weight % of the active material.

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### **EXAMPLE 1**

A liquid fabric softening composition was made as follows.

The cationic fabric softening agent and the fatty acid were premixed and heated together until clear (60-75°C). The molten mixture thus formed was added over a period of at least one minute, via a dip pipe, to water at 45-60°C, with constant stirring to form a dispersion. Other minor ingredients including perfume were added with constant stirring whilst the temperature of the dispersion was greater than 35°C. The nonionic surfactant was most preferably added after the mixture had cooled to 35°C or below.

In this Example, the amounts of component materials used were such that the final product had the following composition expressed as weight %.

Arguad 2HT 3.6%

Pristerene 4916 0.6%

Nonidet LE 6T 0.25%

Perfume 0.13%

Approx. mole ratio of cationic:nonionic 11.1

For comparison purposes Example 1 was repeated except that the Nonidet LE 6T was excluded from the composition (Example 1A).

Both products were assessed visually after recovery from 1,3 and 6 freeze thaw cycles.

A freeze thaw cycle in this context involves storing 100g of product in a screw-capped polyethylene bottle for 16 hours at a temperature of -12°C or less. Such low temperatures are essential to ensure that the products are completely frozen. The product is then allowed to thaw at ambient temperatures for 8 hours.

After 1 freeze thaw cycle Example 1 gave a composition which was slightly thicker than normal. After 3 and 6 freeze thaw cycles Example 1 gave a composition which had normal rinse conditioner rheology ie. was mobile and could be poured in a continuous stream. Example 1A after 1, 3 and 6 freeze thaw cycles gave a composition which was just mobile but which did not pour in a continuous stream.

The results show that the addition of a small amount of a nonionic surfactant to a composition containing a cationic fabric softening agent and a fatty acid gives a product with improved appearance and flow characteristics which are maintained even after 6 freeze thaw cycles.

### **EXAMPLE 2**

This Example demonstrates the effect of replacing Nonidet LE 6T by other nonionic surfactants. Compositions with the following formulations expressed as weight % were prepared by the method previously described.

Arquad 2HT 3.6%

Pristerene 4916 0.6%

Nonionic 0.4%

Perfume 0.13%

For comparison purposes a composition containing no nonionic (Example 2L) was prepared and tested. In Example 2A a level of APG 300 of 0.2% rather than 0.4% was used. Each composition was subjected to the following cap dispenser test after 1 and 3 freeze thaw cycles.

Products were allowed to recover from freezing and a known amount (usually 20 grams) was weighed into an internally screw threaded cap with a total capacity of approximately 25 cm<sup>3</sup> and of known weight. The cap was then inverted over a waste container for 10 seconds and re-weighed. The results obtained are expressed as a percentage of the amount of product weighed into the cap. Prior to freezing, the samples were found to leave up to approximately 10% residue in the cap.

The results were as follows.

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Ex No	Nonionic Surfactant	Cap Dispo		Approx Mole	
	Bullactant	<del></del>	reeze/thaw	ratio of Cationic to	
		cyc:		Nonionic	5
		<u>3,3.</u>	3	HOHEOHEC	
		<del>-</del>	<u>=</u>		
2A	APG 300	69	31	13:1	10
В	DMC AO	27	16	4:1	
С	Dobanol 91-6	34	29	7:1	15
D	Dobanol 45-18	85	45	16:1	
E	Etocas 35	80	45	39:1	
F	Genapol 0-050	83	36	8:1	20
G	Genapol 0-200	59	53	18:1	
H	Nonidet LE 6T	36	36	7:1	
I	Nonyl phenol 2	0EO 24	46	17:1	25
J	Tergitol 15-S-	7 49	38	8:1	
K	Tween 20/Span	20			
	i (50:50)	46	42	10:1	30
L	None	73	68	-	

The results obtained show that the beneficial effect of adding a small amount of nonionic surfactant to a composition containing a cationic fabric softening agent and a fatty acid is retained if the Nonidet LE 6T is replaced by one of a variety of other nonionic surfactants. For some nonionic surfactants the optimum effect is achieved after several freeze thaw cycles.

Similar beneficial results can also be obtained if the Nonidet LE6T is replaced by Tween 20, Alfol 1214-7, Alfol 1214-11, Alfol 1214-13, Brij 30, Brij 35, Lutensol AO7, Lutensol AO8, and Synperonic A11.

## **EXAMPLE 3**

This example demonstrates the effect of increasing levels of nonionic surfactant on the freeze-thaw stability of a 4.2% active with a ratio of Arquad 2HT:Pristerene 4916 of 6:1 by weight and a perfume level of 0.13%. The compositions were prepared by the method previously described. The levels of nonionic are expressed in terms of wt% of the final formulation. The products were assessed after one freeze thaw cycle via the cap dispenser test described above.

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	3A	Etocas 35	0.4	80	39:1
	В	11	1.0	47	16:1
5	С	11	2.0	62	8:1
	D	11 -	4.0	16	4:1
	E	11	6.0	13	3:1
10	F	11	8.0	11	2:1
10	G	APG 300	0.2	69	13:1
	H	11	0.5	57	5:1
15	I	tt	1.0	40	3:1
10	J	11	2.0	22	1:1
	K	11	3.0	17	1:1
20	L	11	4.0	Sep	1:1
20	М	Dobanol 45-18	0.4	85	16:1
	N	11 11	1.0	31	7:1
25	О	11 11	2.0	15	3:1
	P	n · · · · · · · · · · · · · · · · · · ·	4.0	13	2:1
	Q	11 11	6.0	Sep	1:1
30	R	Genapol 0-200	0.4	59	18:1
	S	11 11	1.0	21	7:1
	T	11 11	2.0	12	4:1
<i>35</i>	U	tt 11	4.0	9	2:1
	V	11 11	8.0	Sep	1:1
	W	Nonidet LE 6T	0.1	100	27:1
40	X	11 11	0.25	53	11:1
	Y	tt 11	0.4	36	7:1
	${f z}$	11 11	0.6	33	5:1
45	AA	11 11	0.8	19	3:1
	BB	11 11	1. <sup>i</sup> 0	Sep	3:1

where Sep = Phase Separation

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The results show that high levels of nonionic surfactant, (low mole ratios of cationic to nonionic) promote instability to freeze thaw cycling.

# **EXAMPLE 4**

This example demonstrates the effect of low levels of Nonidet LE 6T on formulations other than those containing 4.2% active and a 6:1 weight ratio of cationic fabric softening agent to fatty acid. The compositions were prepared as described above and the percentage ingredient refers to their weight % in the final product.

Ex No C В 10 ingredient 15 3.2 **DMDHTAC** 3.2 4.8 0.3 0.3 1.2 1.2 HTFA Perfume 0.12 0.12 0.33 0.33 20 Nonidet LE 6T 0.2 0.4 Approx. Mole ratio Cationic to Nonionic 12:1 9:1 25

The compositions were evaluated according to the cap dispenser test as described above.

 Ex No
 % Residues

 4A
 38

 B
 gel

 C
 25

 D
 80

In both cases the addition of the nonionic surfactant was found to improve the appearance and flow characteristics of the product.

## **EXAMPLE** 5

Compositions with the following formulations were prepared as described above. Once again the amount of each ingredient refers to weight % in the final product composition.

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	5H		ı	ı	1	ស	ı	Н	i	ı	0.2
5	5G		t	ì	ı	5	1		ı	0.3	0.2
10	5 F		1	ı	2	1		1	i	ı	0.2
	ਨ ਬ		1	ı	വ	ı	┍┥	ı	ı	0.2	0.2
15	5D		1	2	i	1	ı	ı	-	ı	0.2
20	50		ſ	Ŋ	i	I	ı	1		0.3	0.2
20	5B		Ŋ	í	ı	ı	ı	H	i	ı	0.2
<i>25</i>	5A		2	ı	ı	ı	Į	<del></del> 1	i	0.3	0.2
30											
35									-		
40		o/o		185	7P85	22	4916	acid	E	6Т	
45	Example No	Ingredient	Arquad 2HT	Stepantex Q	Stepantex V	Varisoft 22	Pristerene	Iso Stearic	Lauric acid	Nonidet LE	Perfume
50	EXA	Ing	Arq	Ste	Ste	Var	Pri	Iso	Lau	Non	Per
<i>55</i>						-					

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The compositions were assessed visually after recovery from 1 freeze thaw cycle. In each case the addition of a nonionic surfactant improved the appearance and flow characteristics of the composition. This Example

demonstrates that the beneficial effect of adding nonionic surfactant is not restricted to compositions containing Arquad 2HT and Pristerene 4916.

### **EXAMPLE 6**

In this example the softening performance of the following compositions were examined.

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Example No.			
Ingredient %	6A	6B*	10
· ,			
Arquad 2HT	3.6	3.6	
Pristerene 4916	0.6	0.6	· 15
Dobanol 14-18	0.21	_	
Perfume	0.13	0.13	
			20
mole ratio of			
Arquad 2HT:Dobanol 14-18	30.1	_	
			<i>25</i>

\* comparative example

The softening performance was assessed after one freeze/thaw cycle by subjective evaluation by a panel of expert graders.

The compositions were dispersed in tap water to give dilute aqueous dispersions containing 0.01% of active ingredients. Four pieces of cotton towelling (50 grams) were rinsed in a tergotometer with one litre of the aqueous dispersion at ambient temperature for 5 minutes.

The fabrics were then spun-dry and line dried over night. The compositions were found to give a similar softening benefit. However, after one freeze/thaw cycle the composition containing no nonionic surfactant (Example 6B) was just mobile but did not pour in a continous stream whereas the composition according to the invention was slightly thicker than normal rinse conditioner rheology but could still be poured in a continuous stream.

**NOTES** 

Alfol 1214-7: C<sub>12</sub>-C<sub>14</sub> straight chain alcohol (with an even number of carbon atoms), ethoxylated with an average of 7 ethylene oxide groups per molecule.

Alfol 1214-11: As Alfol 1214-7, but with an average of 11 ethylene oxide groups per molecule.

Alfol 1214-13: As Alfol 1214-7, but with an average of 13 ethylene oxide groups per molecule.

APG 300: Alkyl Polyglycoside based on a C<sub>9</sub>-C<sub>11</sub> alcohol and having an average of 1.6 glycosldic rings per mole of alcohol (supplied as a 51% active solution).

Arquad 2HT: Commercially available form of dihardened tallow dimethyl ammonium chloride (DMDHTAC)(supplied as a 75% active paste).

Brij 30: C<sub>12</sub>-C<sub>14</sub> straight chain alcohol (with an even number of carbon atoms and of natural origin) ethoxylated with an average of 4 ethylene oxide groups per molecule.

Brij 35: As Brij 30, but with an average of 23 ethylene oxide groups per molecule.

DMC AO: Dimethyl coco amine oxide supplied as 30% solution.

Dobanol 91-6: C<sub>9</sub>-C<sub>11</sub> branched chain primary alcohol, ethoxylated with an average of 6 ethylene oxide groups per molecule.

Dobanol 45-18: C<sub>14</sub>-C<sub>15</sub> branched chain alcohol, ethoxylated with an average of 18 ethylene oxide groups per molecule.

Etocas 35: Castor oil ethoxylated with an average of 35 ethylene oxide groups per molecule.

Genapol 0-050: Oleyl alcohol ethoxylated with an average of 50 ethylene oxide groups per molecule.

Genapol 0-200: Oleyl alcohol ethoxylated with an average of 200 ethylene oxide groups per molecule.

Lutensol OA7: C<sub>13</sub>-C<sub>15</sub> branched chain alcohol ethoxylated with an average of 7 ethylene oxide groups per molecule.

Lutensol OA8: As Lutensol OA7, but with an average of 8 ethylene oxide groups per molecule.

Nonidet LE 6T: Similar material to Dobanol 91-6 except that it is topped to remove volatile materials.

Nonyl phenol 20EO: Nonylphenol ethoxylated with 20 ethylene oxide groups per molcule.

Pristerene 4916: Commercially available form of hardened tallow fatty acid (HTFA).

Span 20: Sorbitan monolaurate.

Stepantex Q185: A dialkylethoxymethyl ammonium methosulphate based on soft fatty acid.

Stepantex VP85: A dialkylethoxymethyl ammonium methosulphate based on hard fatty acid.

Synperonic All: C<sub>13</sub>-C<sub>15</sub> branched chain alcohol ethoxylated with an average of 11 ethylene oxide groups per molecule.

Tergitol 15-S-7: C<sub>11</sub>-C<sub>15</sub> secondary alcohol ethoxylated with an average of 7 ethylene oxide groups per molecule.

Tween 20: Polyoxyethylene sorbitan monolaurate containing 20 ethylene oxide groups per molecule.

Tween 20/Span 20: A 50/50 weight % mixture. (50:50)

Varisoft 222: Diamidoquaternary based on soft tallow.

### 15 Claims

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1. A liquid fabric softening composition comprising

i) an aqueous base;

- ii) not more than 8% by weight of a water-insoluble cationic fabric softening agent;
- iii) at least 0.2% by weight of a C8-C24 fatty acid; and

iv) a nonionic surfactant

wherein the mole ratio of the cationic fabric softening agent to the nonionic surfactant is within the range from 40:1 to about 1:1.

- 2. A liquid fabric softening composition according to Claim 1 wherein the nonionic surfactant is selected from:
  - i) alkylene oxide adducts of aliphatic alcohols, and alkyl phenols;
  - ii) amine oxides:
  - iii) alkvl polyglycosides:
  - iv) ethoxylated castor oil;
  - v) sorbitan esters, and ethoxylated derivatives thereof; and
  - vi) mixtures of the foregoing materials.
- 3. A liquid fabric softening composition according to Claim 2 wherein the nonionic surfactant is selected from:
  - i) dimethyl coco amine oxide;
  - ii) nonyl phenol ethoxylated with an average of 20 ethylene oxide groups per molecule;
  - iii) polyoxyethylene sorbitan monolaurate ethoxylated with an average of 20 ethylene oxide groups per molecule:
  - iv) a mixture of polyethylene sorbitan monolaurate ethoxylated with an average of 20 ethylene oxide groups per molecule and sorbitan monolaurate in a 50/50 weight ratio.
  - v) a branched chain primary alcohol having 9 to 11 carbon atoms and ethoxylated with an average of 6 ethylene oxide groups per molecule;
  - vi) a straight chain primary alcohol having 12 to 14 carbon atoms and ethoxylated with an average of 4, 7, 11, 13 or 23 ethylene oxide groups per molecule; and
  - vii) a branched chain primary alcohol having 13 to 15 carbon atoms and ethoxylated with an average of 7, 8 or 11 ethylene oxide groups per molecule.
- 4. A liquid fabric softening composition according to Claim 1 wherein the level of the nonionic surfactant is from 0.1 to 4.5% by weight.
- 5. A liquid fabric softening composition according to Claim 1 wherein the mole ratio of the cationic fabric softening agent to the nonionic surfactant is within the range 18:1 to 3:1.6. A liquid fabric softening composition according to Claim 1 wherein the mole ratio of the cationic
- fabric softening agent to the fatty acid is at least 1:1.

  7. A liquid fabric softening composition according to Claim 1 wherein the composition comprises at
- 7. A liquid fabric softening composition according to Claim 1 wherein the composition comprises at least 1% of the cationic fabric softening and less than 8% of the fatty acid.

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