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(54) Fabric softener composition

(57) The invention relates to a fabric softener composition comprising an aqueous phase comprising a cationic softener and a hydrophobic phase comprising a fragrance. The composition should be shaken prior to use for adapting a dispersion which separates into an aqueous phase and a hydrophobic phase after use. The composition my be used as fabric softener to give a pleasant feel, and to prolong the release of fragrance on fabrics up to 7 days.

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Description

[0001] The present invention relates to fabric softener compositions, in particular to fabric softener compositions comprising an aqueous phase and a hydrophobic phase.

[0002] Fabric softeners for domestic use are primarily designed to give a pleasant feel to fabrics, to impart freshness or a pleasant smell, and to control the static electricity that impairs the comfort of handling and wearing clothes. They are generally aqueous dispersions containing a cationic softener as an active material.

[0003] A known fabric softener composition is disclosed in WO 97/16516 comprising a cationic fabric softener, a fragrance, a non-ionic surfactant, a water-soluble ionisable inorganic salt and water. The fabric softener composition is in the form of an aqueous phase with discrete hydrophobic particles dispersed substantially uniformly throughout the aqueous phase. A problem with aqueous dispersions of this type is that the fragrance is mixed intimately with the other ingredients. Some of the ingredients, e.g. the cationic softener, tend to degrade fragrance over time. Also, the degradation of the fragrance is promoted by the aggressive acidic media, pH < 4, contained in such fabric softener compositions.

[0004] It has now been found that a fabric softener composition comprising fragrance and cationic softener can be produced which is substantially free of the disadvantages referred to above.

[0005] Accordingly, the invention provides in one of its aspects a fabric softener composition having an upper hydrophobic phase comprising a fragrance and a lower aqueous phase comprising a cationic softener.

[0006] Compositions according to the present invention are possessed of numerous advantages. By providing of two distinct phases, the fragrance is separated from aggressive media such as the cationic softener and the acidic aqueous phase which at a pH < 4 may act to degrade the fragrance, and as such, its effect is much longer lasting compared with fragrances employed in aqueous dispersion systems.

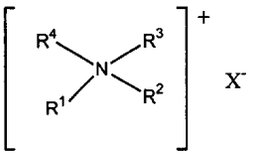
[0007] Further, given that substantially all of the fragrance is located in a hydrophobic phase which sits on the top of the aqueous phase, the user is presented with an enhanced burst of fragrance upon opening a package, e.g. a bottle, containing a composition according to the present invention compared with aqueous dispersion systems wherein the fragrance is dispersed throughout the composition in a less concentrated manner.

[0008] Other advantages of the present invention will be apparent from the following description and examples.

[0009] As the composition is provided as a two-phase system, it should be shaken prior to use to ensure good mixing of all the ingredients. However, because of the problem of fragrance degradation it is desirable that after use, the composition re-establishes its two-phase form as quickly as possible, and preferably separation into the distinct phases occurs within about 10 to about 40 minutes after agitation.

[0010] It has been found that certain softeners promote rapid phase separation. Softeners for use in a composition according to the present invention may be selected from cationic softeners, more particular from the group of cationic quaternary ammonium salts; ester-linked quaternary ammonium compounds; imidazolinium salts; and amido amine salts. Particular examples of these cationic softeners are described in more details below.

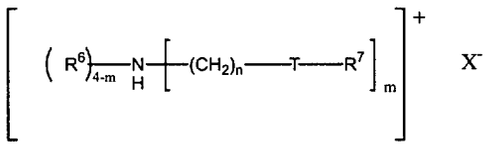
[0011] Quaternary ammonium compounds useful in the present invention may be selected from compounds of the formula:



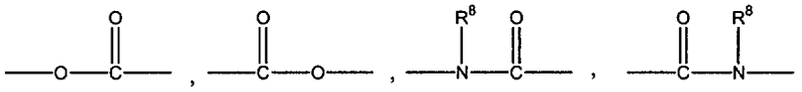
wherein each of R^1 and R^2 is independently selected from a hydrocarbon residue having from 8 to 28, preferably 2 to 24 carbon atoms; R^3 and R^4 represent hydrocarbon residues containing from 1 to 4 carbon atoms; and X^- is an anion, which can be any suitable softener-compatible anion, for example, halide (preferably chloride or bromide), methylsulfate, ethylsulfate, formate, sulfate, nitrate, acetate and the like. Representative examples of these quaternary ammonium compounds include ditallow dimethyl ammonium chloride, ditallow dimethyl ammonium methyl sulphate, dihexadecyl dimethyl ammonium chloride, di(hydrogenated tallow) dimethyl ammonium methyl sulphate, dihexadecyl diethyl ammonium chloride and di(coconut oil alkyl) dimethyl ammonium chloride.

[0012] Suitable materials also include dialkyl ethoxyl methyl ammonium methosulphate based on soft fatty acids, dialkyl ethoxyl methyl ammonium methosulphate based on hard fatty acids, and compounds in which R^3 and R^4 represent methyl, R^2 is $C_{13\text{-}15}$ hydrocarbon group, R^3 is $CH_2CH_2OCOR^5$ where R^5 is stearyl, and X^- is methylsulphate anion.

[0013] Particular preferred cationic softener useful in the composition according to the invention can be an ester-linked quaternary ammonium compound represented by the formula:

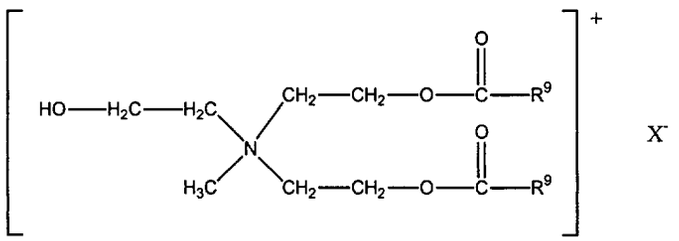


wherein each R^6 group is independently selected from C_{1-6} hydrocarbon residue, such as alkyl, alkenyl or hydroxyalkyl groups; and wherein each R^7 group is independently selected from C_{8-28} linear or branched hydrocarbon residue, such as alkyl or alkenyl groups; T is



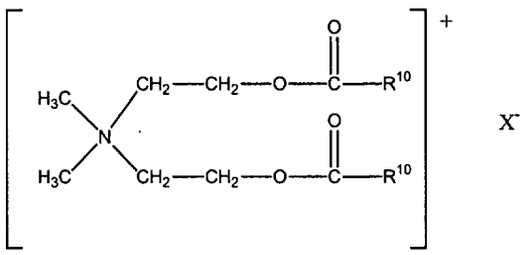
wherein R^8 represents hydrogen, methyl, or ethyl; n is an integer from 0-5 and m is an integer from 1-4; and X^- is an anion as defined as above.

[0014] A preferred material of ester-linked quaternary ammonium compound is that of formula:



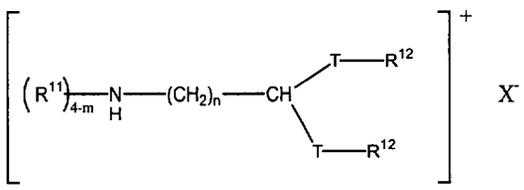
wherein each R^9 is a linear or branched alkyl or alkenyl chain comprising at least 11 atoms such as tallow and X^- is an anion as defined above and, in particular, is methylsulfate. A compound having R^{11} is tallow and X^- is methylsulphate anion is available from Witco under the trade name Rewoquat® WE 18. Another example of this type of material is a compound wherein R^{11} is of partially hardened tallow and X^- is methylsulphate anion.

[0015] Another preferred class of cationic softener containing two ester groups is represented by the formula:



wherein R^{10} is a linear or branched alkyl or alkenyl residue such as tallow, or partially hardened tallow and X^- is an anion as herein above defined and in particular chloride or methylsulphate.

[0016] Other ester-linked quaternary ammonium compounds, which may be used in the compositions of the present invention, are those of the formula:



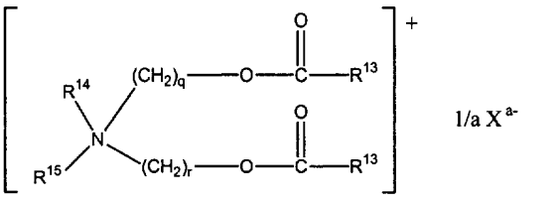
wherein each R^{11} is independently selected from C_{1-4} hydrocarbon residue, such as alkyl, alkenyl or hydroxyalkyl groups; and each R^{12} is independently selected from C_{8-28} linear or branched hydrocarbon residue, such as alkyl and alkenyl groups;

T has the meaning as described above;

n is an integer of from 0-5 and m is an integer from 1-4;

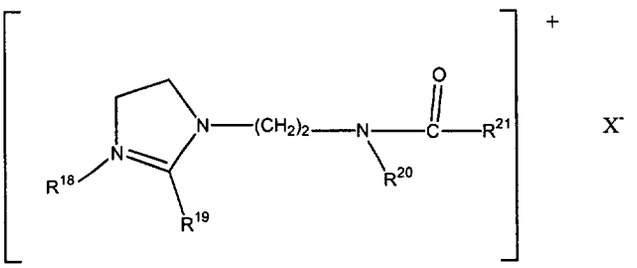
X^- is an anion as defined above, preferably selected from halide, methylsulphate and ethylsulphate.

[0017] The most preferred class of cationic softener is a biodegradable fatty ester quaternary ammonium compound of formula:



wherein each R^{13} independently represents an aliphatic hydrocarbon residue having from 8 to 22 carbon atoms; R^{14} represents $(CH_2)_sR^{16}$ where R^{16} represents an alkoxy carbonyl residue containing from 8 to 22 carbon atoms, benzyl, phenyl, C_1-C_4-alkyl substituted phenyl, OH or H; R^{15} represents $(CH_2)_tR^{17}$ where R^{17} represents benzyl, phenyl, C_1-C_4-alkyl substituted phenyl, OH or H; q,r,s and t each independently, represent a number from 1 to 3; and X^- is an anion as defined above, and the symbol "a" represents the ionic valance of the anion.

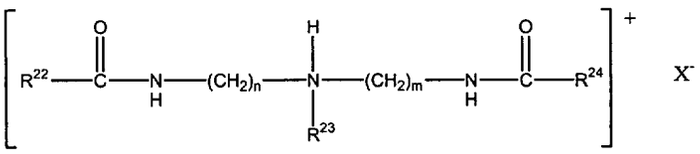
[0018] Other preferred cationic softener are the hydrocarbylimidazolinium salts having the formula:



wherein R^{18} is a hydrocarbon residue containing from 1 to 4, preferably 1 or 2 carbon atoms; R^{21} is a hydrocarbon residue containing from 8 to 25 carbon atoms; R^{19} is a linear or branched hydrocarbon residue containing from 8 to 25 carbon atoms; R^{20} is hydrogen or a hydrocarbon residue containing from 1 to 4 carbon atoms; and X^- is an anion as defined above, and in particular a halide, methylsulphate or ethylsulphate.

[0019] Preferred imidazolinium salts include 1-methyl-1-(tallowylamido-) ethyl-2-tallowyl-4,5-dihydroimidazolinium methosulphate and 1-methyl-1-(palmitoylamido) ethyl-2-octadecyl-4,5-dihydroimidazolinium 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.

[0020] Another class of preferred cationic softener are amido amine compounds of the formula:



wherein R^{22} and R^{23} independently represent C_{12} to C_{30} aliphatic hydrocarbon residues; R^{24} represents $(CH_2CH_2O)_pH$, CH_3 or H; n = 1 to 5; m = 1 to 5; and p = 1 to 10.

[0021] However, of the cationic softener mentioned above, it has been found that the ester-linked quaternary ammonium compounds show a reduced tendency as emulsifying agents compared with the other classes of cationic softener and thereby promote rapid separation of the composition into two distinct phases. Other advantages of these

softeners are the good biodegradibility and their efficient softening results. Because of the aforementioned advantages, ester-linked quaternary ammonium compounds are preferred. Most preferred cationic softeners according to the present invention include Rewoquate® WE18 and Rewoquate® WE15.

[0022] Notwithstanding that the softener referred to hereinabove permit phase-separation within an acceptable time period, nevertheless they are surfactants and it is in the nature of surfactants to resist, to a certain extent, phase-separation and as such it is preferred to use as little cationic softener as possible. However, the cationic softener is needed to impart softness to fabrics and so one has to strike a balance between the need for rapid phase separation and the competitive need for acceptable fabric softness. It is within the realm of routine experimentation for the skilled person to find an appropriate balance having regard to these competing factors. However, it is preferred to use in the composition according to the present invention about 1 % to about 18 % (w/w) of a cationic softener, preferably 2 % to 13 % (w/w).

[0023] Non-ionic surfactants are used in fabric softener compositions of the prior art for example, in combination with cationic softeners. However, we have found that non-ionic surfactants promote emulsification to an extent that phase separation is not quickly established using these materials. Accordingly, it is preferred to avoid the use of non-ionic surfactants in compositions of the present invention.

[0024] Other excipients optionally present in the aqueous phase are anti-foaming agents, thickeners, fluorescent whitening agents, optical brightening agents, anti-wrinkling agents, anti-shrinking agents, sunscreens, colour care agents, colorants, or ironing aids.

[0025] The fabric softener composition according to the present invention comprise up to 8 % (w/w) of these other excipients, preferably from 2 % to 5 % (w/w).

[0026] The hydrophobic phase according to the present of the invention comprises an oil and fragrance.

[0027] To achieve a fast separation after agitation of the composition according to the present invention such oils are preferred wherein the HLB (hydrophilic-lipophilic balance) is below 5, more preferably between 4 and 1. The oil may be a hydrocarbon oil, for example a mixture of hydrocarbon oils obtained from petroleum, in particular petroleum jelly or for example Isopar® produced by Exxon, in particular Isopar® L, Isopar® M or Isopar® H. Other preferred oils are silicone oils such as dimethicone polysiloxane or Tegopren®; or ester oil such as isopropylmyristate. Mixtures of ester oil, hydrocarbon oil and silicone oil may also be used. Because of environmental reasons biodegradable oils are preferred. Accordingly, most preferred oils are esters oils, for example isopropylmyristate, isopropylpalmitate, neopentyl glycol diethylhexanoate or glyceryl triheptanoate. To avoid alteration of the characteristic of the fragrance by the oil, such oils are preferred, which have a low evaporation rate.

[0028] The fabric softener composition according to the present invention comprises 1 % to 50 % (w/w) of the hydrophobic phase, preferably 3 % to 20 % (w/w), and most preferred 5 % to 12 % (w/w).

[0029] In addition to the role of providing a supporting phase for the fragrance which is separate from the aggressive media contained in the aqueous phase, the oil may also treat a fabric to impart softness thereto. Accordingly, the presence of the oil may have the beneficial effect of permitting the use of less cationic softener which is relatively expensive compared with the oil.

[0030] Further, the oil may coat the fragrance, such that when the composition is applied to a fabric it tends to promote prolonged release of fragrance and so increasing the substantivity of the fragrance on the fabric, for example preferred compositions according to the invention may impart fragrance to a fabric for up to 7 days after treatment. It was found that the rinse loss of the fragrance during the rinse cycle and/or the fragrance lost during the tumble drying process is much lower compared with aqueous dispersion fabric softeners. On dry fabrics a 3 - 5 time stronger fragrance, measured by headspace, can be achieved compared with conventional aqueous dispersion fabric softeners. The headspace analysis is described in the Journal of Agriculture and Food Chemistry, Vol. 19, No. 6 (1971), page 1049 - 1056.

[0031] The composition according to the present invention may contain all kinds of fragrances of natural and/or synthetic origin, for example geraniol, citronellyl acetate and vanilline. The composition comprises one fragrance or a mixture of fragrances. However, to limit the migration of fragrance from the hydrophobic phase to the aqueous phase water soluble fragrances, for example phenylethylalcohol and diethylmalonate are preferably to be avoided. Accordingly, preferred fragrances have a clogP greater than 2, most preferred a clogP greater than 3. In a preferred embodiment the composition according to the present invention comprises about 0.1 % to about 3 %(w/w) fragrance. Most preferred are fragrance compositions having at least 80 % (w/w) of fragrance with a clogP greater than 2.

[0032] Optionally, the hydrophobic phase may comprise a lipophilic dye, for example Puricolor® blue FBL5, Puricolor® blue FBL5 Puricolor® blue ABL9, Puricolor® green U3 or Puricolor® yellow AYE23 from Ciba.

[0033] Particularly preferred composition according to the present invention comprises from 2 % to 15 % (w/w) cationic softener; from 3 % to 20 % (w/w) ester oil; from 0.1 % to 3 % (w/w) fragrance; from 57 % to 92.9 % (w/w) water; and from 2 % to 5 % (w/w) other excipients.

[0034] Compositions according to the present invention may be used as fabric softeners. They may be added to the last rinse cycle in a amount of 35 ml to 150 ml per 4 kg of fabrics in general, or garments in particular. However, the amounts used is based upon the preferences of the user.

[0035] The invention is illustrated by the following examples.

Example1

[0036] A fabric softening composition of the present invention (herein after named as "bi-phase fabric softener") was formulated as follows:

		% w/w
Part A		
Rewoquat® WE 18 (Goldschmidt)	di-(tallow carboxyethyl) hydroxy-ethyl methylammonium methosulfate	10.00
Deionised Water		74.45
Calcium chloride		0.50
Myacide® BT330 (Boots)	2-bromo-2-nitrophropane-1,3-diol	0.05
Part B		
Isopropylmyristate		11.50
Isopar ® M (Exxon)		2.50
Fragrance		1.00
		100.00

[0037] The water and the Rewoquat® WE 18 was heated separately to 50°C. While stirring the preheated water, one half of the Rewoquat® WE 18 (preheated to 50°C) was added. Calcium chloride was added and then the remaining part of the Rewoquat® WE 18 while stirring the mixture. After cooling to room temperature under stirring, Myacide® BT330 was added. Finally, part B was added.

Example 2

Fragrance evaluation: substantivity profile

[0038] The bi-phase fabric softener composition prepared according to example 1 was compared to a mono-phase fabric softener composition formulated as follows:

		% w/w
Part A		
Deionised Water		81.75
$MgCl_2$ (saturated solution)		1.0
Part B		
Rewoquat® WE 18 (Goldschmidt)	di-(tallow carboxyethyl) hydroxy-ethyl methylammonium methosulfate	13.0
GenapolO100 (Hoescht)	Ethoxylated fatty alcohol C16 - C18 10EO	2.0
Antifoam 110A-EU (Dow Corning)	silicone emulsion	1.0
Part C		
Dequest 2010 (Monsanto)	Hydroxyethyliden 1,1 diphosphonic acid	0.2
Proxel GXL (ICI)	Benzisothiazolinone sodium salt	0.02
Bronidox L (Cognis)	5 bromo-5nitro-1,3 Dioxane	0.03
Fragrance		1.0
		100.00

[0039] While stirring part A was heated to 65°C. Then part B (preheated to 65°C) was added while stirring the mixture. After cooling to room temperature part C was added.

[0040] A European washing machine was loaded with 4 kg mixed fabrics, including cotton terry towels. In the last rinse cycle 35 grams of the fabric softener was added. The cotton terry towels were used to asses the performance of each fabric softener by an expert panel.

Table 1

	Bi-phase fabric softener	mono-phase fabric softener
dry towels after 24 hours	4	3
dry towels after 5 days	4	2

Evaluation scale:
5 = very strong 4 =strong 3 = moderate 2 = weak 1 = very weak

[0041] Panellists compared the strength of the fragrance on dry fabrics (Table 1). A much more improved fragrance strength and fragrance quality was achieved on dry fabrics with the bi-phase fabric softener composition prepared according to example 1 compared with the mono-phase fabric softener composition prepared according to the formulation above. Furthermore, the fragrance appears fresher using the bi-phase fabric softener.

Example 3

Fragrance evaluation: Headspace

[0042] The cotton terry towels were washed according to the washing protocol of example 2. The towels were line dried and placed in a glass container 1 day / 5 days after drying. During 15 minutes and an extraction flow of 30ml/minute the headspace was collected. Then the samples were analysed by GC/MS. The results are shown in Table 2.

Table 2

	bi-phase fabric softener	mono-phase fabric softener
dry towels after 24 hours	489ng/L	148ng/L
dry towels after 5 days	3g4ngIL	89ng/L

[0043] The bi-phase fabric softener delivered significantly more fragrance on the cotton terry towels.

Example 4

Fragrance composition

[0044]

Ingredients	% w/w
Benzyl Acetate	4.0
Bornyl Acetate	4.0
Citronellyl Acetate	0.8
Menthanyl Acetate	2.0
PT Butylcyclohexyl Acetate	3.0
2-Propenyl Phenoxyacetate	3.6
Phenyl Ethyl Acetal	1.0
Verdyle Acetate ™	10.0
Cyclamen Aldehyde Extra	2.5
Benzyl Benzoate	8.0
Citronellol	16.0
Coumarine pure Crist.	7.0
Diphenyl Oxyde	2.0

(continued)

Ingredients	% w/w
Geraniol 980	4.0
Isoraldeine® 70	5.2
Lavandin Grosso Ess Orpur	2.0
Lilial	3.5
Linalool Synthetic	2.0
N-112	0.1
Peche Pure®	0.1
Rosacetol™	7.5
Terpineol Pure	10.0
Vanilline	1.0
Virdine®	0.7
	100.0

Claims

1. A fabric softener composition having an upper hydrophobic phase comprising a fragrance and a lower aqueous phase comprising a cationic softener.

2. A fabric softener composition according to claim 1 wherein the cationic softener is selected from the group of imidazolinium salts; amido amines; and ester-linked quaternary ammonium salts.

3. A fabric softener composition according to claim 1 and 2 comprising up to 18% (w/w) of the cationic softener.

4. A fabric softener composition according to any of the preceding claims wherein the oil is selected from the list of hydrocarbon oil, silicone oil and ester oil.

5. A fabric softener composition according to claim 4 comprising hydrocarbon oil, silicone oil or ester oil with a hydrophilic-lipophilic balance less than 5.

6. A fabric softener composition according to any of the preceding claims comprising from 1% to 50% (w/w) of the oil.

7. A fabric softener composition according to claim 1 comprising

from 2% to 18% (w/w) of cationic softener;
from 1% to 50% (w/w) of oil;
from 0.1% to 3% (w/w) of fragrance; and
from 29% to 96.9% (w/w) of water.

8. A fabric softener composition according to any preceding claims wherein the fragrance is **characterised by** a clogP greater than 2, preferably a clogP greater than 3.

9. A fabric softener composition adapted to disperse upon agitation, wherein the dispersion separates into a hydrophobic phase and an aqueous phase from about 10 to about 40 minutes after agitation.

10. A method of prolonging the release of fragrance from a fabric up to 7 days comprising the step of treating a fabric with a composition as described in any of the preceding claims.

11. A packaged fabric softener composition comprising

from 2% to 18% (w/w) of cationic softener;
from 1% to 50% (w/w) of oil;
from 0.1% to 3% (w/w) of fragrance; and

from 29% to 96.9% (w/w) of water.

12. A packaged fabric softener composition **characterised by** a upper hydrophobic phase comprising a fragrance and a lower aqueous phase comprising a cationic softener.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 01 11 8341

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.7)
X A	DE 200 14 021 U (HENKEL KGAA) 11 January 2001 (2001-01-11) * examples 9-11 * * page 5, last paragraph - page 6, paragraph 3 * * page 7, paragraph 1 * * page 13, paragraph 4 * * page 14, last paragraph - page 15, paragraph 2 *	1,2,12 3-11	C11D17/00 C11D3/50 C11D3/18 C11D3/37 C11D3/20 C11D1/62
X	WO 95 22594 A (UNILEVER PLC) 24 August 1995 (1995-08-24) * examples 9,10 * * page 1, line 1 - line 7 *	11	
X	EP 0 371 534 A (UNILEVER NV) 6 June 1990 (1990-06-06) * example III * * abstract *	11	
X	EP 0 829 531 A (UNILEVER PLC) 18 March 1998 (1998-03-18) * examples A,C-H * * page 6, line 28 - line 31 *	11	TECHNICAL FIELDS SEARCHED (Int.Cl.7) C11D
X	WO 99 43777 A (UNILEVER PLC) 2 September 1999 (1999-09-02) * examples 12-15 * * Table 2: composition with oil:cationic active ratio of 1.4:1 * page 1, line 8 - line 13 *	11	
X	US 6 251 850 B1 (CREUTZ SERGE FIRMIN ET AL) 26 June 2001 (2001-06-26) * examples 1,2 * * column 4, line 27 - line 64 * * column 5, line 64 - column 6, line 2 *	11	

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	15 February 2002	Neys, P

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
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D : document cited in the application
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Application Number
EP 01 11 8341

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.7)
A	US 3 533 955 A (MARTIN DONALD JAY ET AL) 13 October 1970 (1970-10-13) * claims 1,2,5-8 * * examples 1-29,35-37 * * column 2, line 5 - line 14 * * column 4, line 60 - line 67 *	1-12	
A	DE 199 45 503 A (HENKEL KGAA) 5 April 2001 (2001-04-05) * claims 1,2,4,5,11,12,23 * * example E1 * * page 3, line 43 - line 68 * * page 5, line 65 - line 66 *	1-9,11, 12	
			TECHNICAL FIELDS SEARCHED (Int.Cl.7)

The present search report has been drawn up for all claims

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THE HAGUE	15 February 2002	Neys, P

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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
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L : document cited for other reasons

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