

(19)



Europäisches Patentamt

European Patent Office

Office européen des brevets



(11)

**EP 0 829 531 A1**

(12)

**EUROPEAN PATENT APPLICATION**

(43) Date of publication:

**18.03.1998 Bulletin 1998/12**

(51) Int Cl.<sup>6</sup>: **C11D 3/00**, C11D 1/62,

C11D 3/18, C11D 3/20,

C11D 3/43, C11D 3/50,

C11D 1/72

(21) Application number: **97306076.7**

(22) Date of filing: **08.08.1997**

(84) Designated Contracting States:

**AT BE CH DE DK ES FI FR GB GR IE IT LI LU MC  
NL PT SE**

(30) Priority: **22.08.1996 GB 9617612**

(71) Applicants:

- **UNILEVER PLC**

**London EC4P 4BQ (GB)**

Designated Contracting States:

**GB IE**

- **UNILEVER N.V.**

**3013 AL Rotterdam (NL)**

Designated Contracting States:

**BE CH DE DK ES FI FR GR IT LI LU MC NL PT SE  
AT**

(72) Inventors:

- **Mohammadi, Mansur Sultan**

**Bebington, Wirral, L63 9NA (GB)**

- **Moorfield, David**

**Warrington, Cheshire, WA4 3BB (GB)**

- **Sams, Philip John**

**South Wirral, Cheshire, L64 1TB (GB)**

- **Tollerton, Sigrun**

**Wrexham, Wales, LL11 3DW (GB)**

(74) Representative: **Tansley, Sally Elizabeth et al**

**Unilever PLC,**

**Patent Division,**

**Colworth House**

**Sharbrook, Bedford MK44 1LQ (GB)**

(54) **Fabric conditioning composition**

(57) A fabric conditioner that easily disperses in water and softens without effecting the water absorbency of the fabrics treated therewith. The fabric conditioner

comprises a cationic fabric softening compound and an oil in which the cationic fabric softening compound is suspended in the oil.

**EP 0 829 531 A1**

**Description**Technical Field

5 The present invention relates to fabric conditioning compositions. In particular the present invention relates to fabric conditioning concentrates with less than 10% by weight of water that easily disperse and self-emulsify in cold water (10-25°C) and when emulsified give excellent perfume delivery and softening to laundry.

Background and Prior Art

10 Conventional rinse conditioners are obtained by dispersing a cationic softening material and perfume into hot water. The problem with such conventional rinse conditioners is that although the rinse conditioners soften laundry they do not deliver perfume onto the fabric well because as much as one third of the perfume in the formulation remains in the rinse water.

15 Rinse conditioners based on emulsions are known. We have found that such emulsion-based softeners perfume fabrics more effectively than these conventional rinse conditioners but subsequently have a loss in their softening performance.

20 WO 92/18593 (Procter and Gamble) discloses a granular rinse conditioner, which can be added to water to form an aqueous dispersion, comprising a fatty alkyl ester of a polyhydric alcohol as the softening compound and a mono-long chain alkyl cationic surfactant.

EP 404 474 (Unilever) discloses a clear, isotropic fabric softening compound comprising a cationic fabric softening material and a carboxylic acid having a total number of 8 carbon atoms.

25 GB 2 007 734 (Cargo Fleet) discloses a liquid fabric softener concentrate which consists of a quaternary ammonium salt having at least one C<sub>8</sub>-C<sub>30</sub> long chain alkyl group and an oil. The concentrate can be dispersed or emulsified with water. No mention of improved perfume delivery is made.

The present invention overcomes the problems associated with the prior art in that it provides a rinse conditioner with excellent perfume delivery and fabric softening properties.

30 Additionally the present invention also provides rinse conditioners which are not detrimental to the absorbency of fabric and which also reduce the creasing of fabric.

Definition of the Invention

The present invention relates to a conditioning concentrate comprising a cationic fabric softening compound and oil in which the cationic fabric softening compound is suspended in the oil.

35 The invention also relates to the use of a composition described above during the rinse cycle to reduce creasing or to improve the water absorbency of fabric.

A process for perfuming and softening laundry is also described in which the composition described above is added directly to the rinse liquor.

Detailed description of the invention

40 The present invention relates to a conditioning concentrate. The term concentrate in the context of the present invention means that little or no water is present in the formulation. The maximum level of water that can be present in the formulation is 10 % or less by weight of the total formulation, more preferably 5 % or less by weight, most preferably 2 % or less by weight. In some situations less than 0.5% by weight of water may be present.

45 It is especially advantageous if the softening compound and the oil are heated together to form a melt. Perfume may also form part of the melt.

50 Without being bound by theory the compositions of the invention can be described as having a physical state wherein a network of solid crystallites of controlled strength is formed to contain the liquid phase. The strength of the solid network is controlled such that composition does not undergo gravitational sedimentation under quiescent conditions but flows under agitation and stirring. In direct contrast a conventional emulsion comprises an oil emulsified by cationic active in the form of lamellar layers, said emulsion can yield to gravitational phase separation.

Further discussion on the properties of inorganic solids suspended in an oil are given in "Electrostatic Stabilization of Suspensions in Non-aqueous Media" Ph.C van der Hoeven, University of Wageningen Thesis (1991), Chapter 2.

55 It is preferred that when added to water the emulsified product has an oil droplet size ( $D_{43}$  volume average droplet size) of under 5µm and more preferably under 3µm. Droplet size ( $D_{10}$ ) is typically in the range 0.2µm-50µm.

The Oil

The compositions of the present invention comprise at least one oil. The oil may be a mineral oil, an ester oil or a sugar ester oil. Some natural oils, such as vegetable oils may be included if appropriate.

It is preferred if the oil is an ester oil, sugar ester oil or a mineral oil. Suitable oils include those in the Sirius range of mineral oils (ex Silkolene).

Suitable ester oils include the saturated ester oils (ex Unichema) and the unsaturated sugar ester oils (ex Mitsubishi Kagaku).

It is preferred if the ester oils of the invention are hydrophobic in nature. It is further preferred if the ester oil is saturated (hardened) in nature, unless it is a sugar ester oils for which unsaturation is preferred.

Suitable ester oils are the fatty ester of a mono or polyhydric alcohol having from 1 to about 24 carbon atoms in the hydrocarbon chain, and mono or polycarboxylic acids having from 1 to about 24 carbon atoms in the hydrocarbon chain with the proviso that the total number of carbon atoms in the ester oil is equal to or greater than 16 and that at least one of the hydrocarbon radicals in the ester oil has 12 or more carbon atoms.

Ester oils most suitable for use in the present invention are the PRIOLUBES from Unichema. In particular PRIOLUBE 1407, PRIOLUBE 1447, PRIOLUBE 1415, PRIOLUBE 1446, PRIOLUBE 1427, PRIOLUBE 1445, PRIOLUBE 2045, PRIOLUBE 3988, PRIOLUBE 3987, PRIOLUBE 2091, ESTOL 1545 and ESTOL 1527 are advantageously employed. Of these PRIOLUBE 2045, which is a neopentyl glycol monomerate, PRIOLUBE 1446, which is a neopentyl glycol dioleate, and Estol 1445, which is a 2-ethyl hexyl stearate are particularly useful. The fatty acid mixture for this ester is called in the oleochemical industry "monomer fatty acid" and derives from the dimerisation of rape oil (eruca low) fatty acid or oleine from tallow. In the dimerisation process, dimer, trimer acids and so called monomeric acids are formed. After the dimerisation the "monomeric" part is separated via distillation.

Suitable mineral oils include Esso Marcol technical grade range of oils and particularly preferred is the Silkolene medicinal Sirius range.

The molecular weight of the mineral oil is typically within the range 150 to 400.

It is preferred if the viscosity of the ester oil or mineral oil is from 2 cP (mPa.s) to 400 cP (mPa.s) at a temperature of 25°C, more preferably a viscosity from 2 to 150 cP (mPa.s), most preferably a viscosity from 10 to 100 cP (mPa.s).

It is preferred if the viscosity of the sugar ester oil is above 50,000 cP, preferably 5,000 to 20,000 cP, most preferably 6,000 to 20,000 cP. All viscosities are measured at 25°C.

It is preferred if the density of the mineral oil is from 0.80 to 0.90 g/cm<sup>3</sup>, more preferably from 0.83 to 0.88g/cm<sup>3</sup>.

It is further preferred if the refractive index of the oil is from 1.445 to 1.490, more preferably from 1.460 to 1.485.

The level of oil in the rinse conditioner is preferably from 20 to 80 wt% of the composition, most preferably from 50 to 70 wt%.

The Crystal Growth Inhibitor

It is also preferred if a crystal growth inhibitor is present. It is beneficial if the crystal growth inhibitor forms part of the melt.

The crystal growth inhibitors are compounds that have highly polarisable hydrophilic groups.

Examples of suitable crystal growth inhibitors include (poly) carboxylates, fatty acids, (poly) ethylene oxides, hydroxylic organic acids, (poly) phosphates, organic phosphonates, amino phosphonates, poly acrylic acids, poly aspartic acid, poly propylene glycols, polyethylene glycols and soil release polymers such as PET-POET (PERMALOSE TM ex ICI), SOKOLAN HP 22 (ex BASF), cationic decoupling polymers (ex National Starch) as disclosed in (EP 0 415 69842).

Particularly preferred crystal growth modifiers are organic acids of alkyl chain length C<sub>18</sub> or less, and nonionic surfactants having an average alkyl chain length between C<sub>10</sub> and C<sub>22</sub> and from 10 to 30 ethoxylate groups. Particularly preferred crystal growth inhibitors are tallow and coco nonionic surfactants having from 15 to 20 ethoxylate groups, organic acids such as lactic acid (which contains about 20% linear polymeric self-esterified esters), stearic acid, and hardened or unhardened tallow acid. Mixtures of crystal growth inhibitors may also be used.

When the compositions of the present invention are being produced by the melt process as herein described it is particularly preferred that the compositions comprise a fatty acid crystal growth modifier, especially hardened or non-hardened tallow fatty acid or lactic acid or a tallow ethoxylated alcohol.

If the crystal growth inhibitor is a fatty acid or nonionic surfactant it is advantageous if the alkyl chain is not branched.

The average alkyl chain length of the nonionic surfactant may, for example be between C<sub>10</sub> to C<sub>14</sub>. If a nonionic surfactant is used as the crystal growth inhibitor it is preferred if the HLB is from 14 to 17 especially from 15 to 17.

Preferred crystal growth inhibitors have further advantages in that they control crystal agglomeration (and hence the viscosity) and aid emulsification of the oil. Furthermore, the preferred crystal growth inhibitors aid the dispersion of the melt in water.

The crystal growth inhibitor can be cationic and nonionic in nature but not anionic. In this context fatty acids if used (in presence of some water) as a crystal growth inhibitor should be used at a pH where they are not dissociated and are thus not anionic in nature.

The crystal growth inhibitor or mixtures thereof should preferably be present at a level of from 1 wt% to 20 wt% of the composition, more preferably the crystal growth inhibitor should be present at a level from 2 wt% to 10 wt%. As described above a mixture of crystal growth inhibitors may be used, however the level of each individual crystal growth inhibitor is preferably between 1 wt% and 10 wt%, more preferably between 1 wt% and 6 wt% of the total composition.

It is preferred that the compositions comprise a co-emulsifier for rapid dispersion of the composition when it is added to water.

If a nonionic ethoxylated surfactant is used as the crystal growth inhibitor then this will function as both the inhibitor and as a co-emulsifier to provide good dispersion.

It is a preferred feature of the present invention that the compositions comprise a co-emulsifier which is a surfactant having a low HLB, preferably less than 14. It has been found that such surfactants provide excellent dispersion results.

The preferred co-emulsifiers are  $C_8$ - $C_{22}$  alcohol alkoxylates with an average of 3 to 10 alkoxylate groups, preferably 5 to 7 alkoxylate groups. Ethoxylates are the preferred alkoxylates although mixed ethoxylates/propoxylates or propoxylates may also be used.

Alternatively, the compositions may comprise, as the co-emulsifier, a mixture of surfactants to provide good dispersibility. A mixture of a nonionic ethoxylate surfactants having an HLB of less than 14 with surfactants having an HLB of greater than 15.5 also provides excellent dispersibility in water.

Suitable surfactants with an HLB of less than 14 are disclosed hereinabove. Suitable surfactants with an HLB of greater than 15.5 include  $C_8$ - $C_{22}$  alcohol alkoxylates with an average of 15 to 25 alkoxylate groups, preferably 17 to 23.

Again ethoxylates are preferred, although mixed ethoxylates/propoxylates and propoxylates may be used.

The co-emulsifier mixture preferably comprises no more than 90% by weight of the higher HLB surfactant, preferably no more than 75%, especially no more than 65%.

#### The Fabric softening Compound

The compositions of the present invention comprise at least one fabric softening compound.

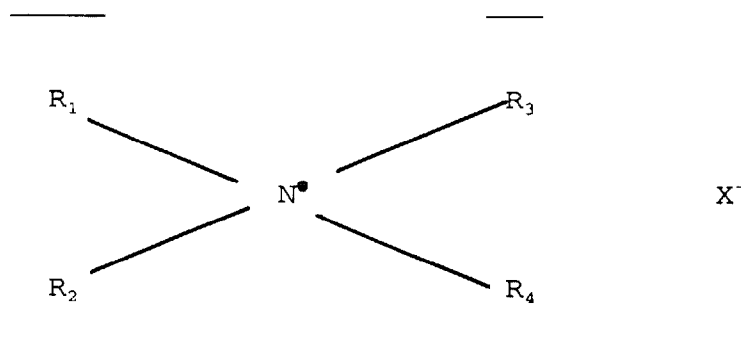
The fabric softening compound is preferably a quaternary ammonium material comprising a polar head group and two alkyl or alkenyl chains.

Preferably the fabric softening compound of the invention has two long chain alkyl or alkenyl chains with an average chain length greater than  $C_{14}$ , more preferably each chain has an average chain length greater than  $C_{16}$ , more preferably at least 50% of each long chain alkyl or alkenyl group has a chain length of  $C_{18}$ .

It is preferred if the long chain alkyl or alkenyl groups of the fabric softening compound are predominantly linear.

It is highly preferred if the fabric softening compounds of the invention are substantially water insoluble. Substantially insoluble fabric softening compounds in the context of this invention are defined as fabric softening compounds having a solubility less than  $1 \times 10^{-3}$  Wt% in demineralised water at 20°C, preferably the fabric softening compounds have a solubility less than  $1 \times 10^{-4}$ , most preferably the fabric softening compounds have a solubility at 20°C in demineralised water from  $1 \times 10^{-8}$  to  $1 \times 10^{-6}$ .

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

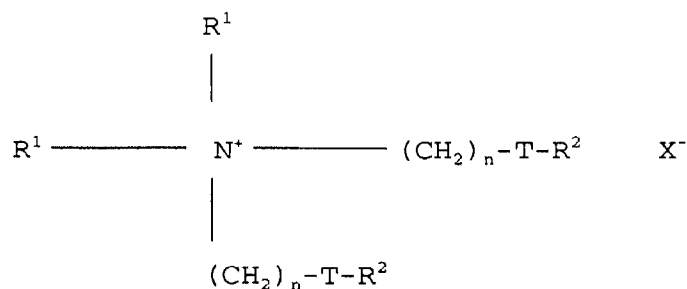


wherein  $R_1$  and  $R_2$  represent hydrocarbyl groups having from 12 to 24 carbon atoms;  $R_3$  and  $R_4$  represent hydrocarbyl groups containing 1 to 4 carbon atoms; and X is an anion, preferably selected from halide, methyl sulphate and ethyl sulphate radicals are preferred.

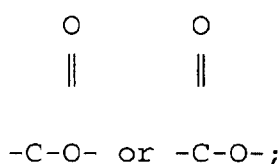
Representative examples of these quaternary softeners include di(tallow alkyl)dimethyl ammonium chloride; di(tallow alkyl) dimethyl ammonium methyl sulphate; dihexadecyl dimethyl ammonium chloride; di(hydrogenated tallow alkyl) dimethyl ammonium chloride; dioctadecyl dimethyl ammonium chloride; di(hydrogenated tallow alkyl) dimethyl ammonium methyl sulphate; dihexadecyl diethyl ammonium chloride; di(coconut alkyl) dimethyl ammonium chloride. Ditalow alkyl dimethyl ammonium chloride, di(hydrogenated tallow alkyl) dimethyl ammonium chloride, and quats of this.

Other preferred softeners contain esters or amide links, for example those available under the tradenames Accosoft 580, Varisoft 222, and Stepanex.

Particularly preferred fabric softening compounds are a water insoluble quaternary ammonium materials which comprises a compound having two C<sub>12-18</sub> alkyl or alkenyl groups connected to the molecule via at least one an ester link. It is more preferred if the quaternary ammonium material has two ester links present. The preferred ester-linked quaternary ammonium material for use in the invention can be represented by the formula:



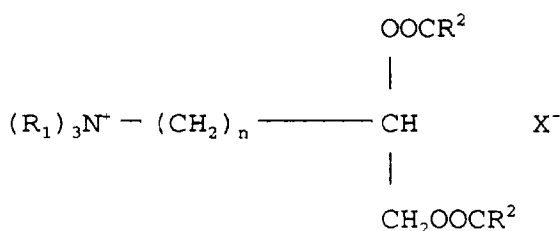
wherein each R<sup>1</sup> group is independently selected from C<sub>1-4</sub> alkyl, hydroxyalkyl or C<sub>2-4</sub> alkenyl groups; and wherein each R<sup>2</sup> group is independently selected from C<sub>8-28</sub> alkyl or alkenyl groups; T is



X<sup>-</sup> is any suitable anion and

n is an integer from 0-5.

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



wherein R<sub>1</sub>, n, X<sup>-</sup> and R<sub>2</sub> are as defined above.

It is advantageous for environmental reasons if the quaternary ammonium material is biologically degradable.

Preferred materials of this class such as 1,2 bis[hardened tallowoyloxy]-3- trimethylammonium propane chloride 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-hardened tallowoyloxy -2-hydroxy trimethylammonium propane chloride.

The fabric softening agent may also be a polyol ester quat (PEQ) as described in EP 0 638 639 (Akzo).

The level of cationic softening compound is preferably from 3 wt% to 60 wt% of the total composition, more pref-

erably from 10 wt% to 40 wt%.

It is preferred if the ratio of cationic softening compound to oil is from 1:10 to 5:1 preferably from 1:5 to 1:1, and most preferably 1:3 to 1:1.

It is preferred that the composition contains less than 25 wt% of the total composition of organic solvent, more preferably less than 20 wt%, most preferably less than 10 wt%.

It is especially preferred that the solvents are non-aqueous. In any case level of water must be kept below 10% of the total composition.

For compositions produced by the melt process, as herein below described it is preferred that organic solvents are included in the compositions. It is preferred that less than half of the amount of any solvent present is flammable solvent (i.e. has a flash point of less than 25°C. The major proportion of the solvent should most preferably be a non-flammable solvent (i.e. have a flash point of higher than 25°C). Suitable examples include IPA, propylene glycol, and especially hexylene glycol and butyl digol for reasons of viscosity and appearance of the melt. A mixture of solvents may provide advantageous results, especially with respect to viscosity. In some compositions solvent may be present as a result of being a component of an ingredient of the composition.

It is believed that the choice of the type of any solvent present in the compositions of the present invention help to control the size of the crystals of the fabric softening compound.

An excess of solvent in the compositions, e.g. greater than 30% by weight of solvent typically produces an increase in the particle size of the fabric softening compound. However, this typically results in compositions which are more difficult to disperse.

#### Composition pH

The compositions of the invention when dispersed in water at use concentration preferably have a pH of more than 1.5, more preferably less than 5.

#### Product Form

The composition is in the form of a concentrate, typically in the form of a paste or high viscosity liquid. The concentrate may be added either immediately or after standing, following addition to water by the consumer to form an emulsion which is then added to the rinse liquor.

However, it is preferable if the concentrate is added directly to the rinse liquor.

#### Other Ingredients

The composition can also contain one or more optional ingredients, selected from pH buffering agents, perfumes, perfume carriers, fluorescers, colorants, hydrotropes, antifoaming agents, antiredeposition agents, polymeric or other thickening agents, enzymes, optical brightening agents, opacifiers, anti-shrinking agents, anti-wrinkle agents, anti-spotting agents, germicides, fungicides, anti-corrosion agents, drape imparting agents, antistatic agents and ironing aids.

The concentrated compositions according to the present invention may be produced according to any suitable method. Two methods are particularly preferred namely the melt process and the cold grinding (milling) process.

A melt as referred to herein is a homogeneous liquid mixture of two or more substances that would individually solidify on cooling to ambient temperature. In our melts typically one or two of the components solidify on cooling (usually cationic and nonionic).

In the melt process the fabric softening compound is heated until it is mobile, preferably liquid, followed by addition to an oil phase (which may contain a perfume) to produce a melt. The additional components of the composition may be incorporated into the composition via a mobile fabric conditioning compound, via the oil, or they may be added after the fabric conditioning compound and the oil have been mixed together. Typically the melt is formed at a temperature of at least 35°C, preferably of at least 40°C, e.g. at a temperature of 45°C to 70°C. Preferably the fabric softening compound and nonionic(s) are mixed with the oil and then heated to form a liquid to which upon cooling to a lower temperature perfume may be added.

Alternatively, the compositions of the present invention may be produced by a cold grinding (milling) method wherein the fabric softening compound and the oil are mixed together, at ambient temperature typically at high shear rates, without the fabric softening composition being heated prior to mixing.

The cold grinding process typically produces compositions of lower viscosities than the corresponding compositions produced by the melt route.

The invention will now be illustrated by reference to the following non-limiting Examples. Further modifications within the scope of the present invention will be obvious to the skilled man.

Comparative Examples are illustrated by a letter and Examples of the invention by a number:

EXAMPLES

5     Examples 1 to 5 and A to H

Examples 1 to 5 were prepared by heating the cationic softener until liquid and adding it to a blend of oil and perfume or adding the cationic to the oil and heating the mixture. Additional components were added to the oil and warmed or added to the cationic softener and warmed or added to the blend of cationic and oil while cooling. This method is referred to herein as the melt process.

Examples A to H were prepared by stepwise addition of components to hot water.

15

20

25

30

35

40

45

50

55

Table 1

Component % w/w	1	A	2	B	3	C	4	D	5	E	F	G	H
n-Hexadecane	66.4	13.3	-	-	-	-	-	-	-	-	-	-	-
Estol 1545			68.0	13.6	66.9	13.4	-	-	-	-	-	-	-
Marcol 52			-	-	-	-	64.9	13.0	-	-	-	-	-
Sirius M70			-	-	-	-	-	-	66.9	13.4	-	-	3.38
Arquad 2HT	27.4	5.5	29.2	5.8	27.9	5.8	27.9	5.6	27.9	5.8	2.13	2.13	2.13
Perfume	6.3	1.3	-	-	4.5	0.9	4.5	0.9	4.5	0.9	0.24	0.24	0.24
Lactic Acid	2.8	0.6	2.8	0.6	-	-	2.7	0.5	-	-	-	-	-
Sirius M180	-	-	-	-	-	-	-	-	-	-	3.38	-	-
Ryoto Sugar Ester	-	-	-	-	-	-	-	-	-	-	-	3.38	-
Coco 20E0	-	-	-	-	0.7	0.14	-	-	0.70	0.14	-	-	-
Water		to 100		to 100		to 100		to 100		to 100	to 100		
Softness Score	4.2	5.2	4.0	4.7	4.3	6.0	4.3	4.8	3.8	5.2	4.7	-	4.67
Perfume Score		2.6			3.60	2.60					3.53	3.69	3.33

Arquad 2 HT is di methyl di hardened tallow quaternary ammonium chloride containing about 20% isopropyl alcohol and was used as supplied ie. the % refers to the product as supplied.

Examples F,G and H are 5% emulsions prepared from the melts by diluting in 50°C water. The perfume score is noted as perfume on "wet" fabric.

Table 2

Oil	Type	Viscosity 25°C/mPa.s	RI 20°C	Density 20°C
n-hexadecane	hydrocarbon (ex Baker	5.98	1.43453	0.778
Estol 1545 2 - ethylhexyl stearate	ester oil (ex Unichema)	13.37	1.44811	0.860
Marcol 52	mineral (ex Esso)	14.49	1.45246	0.829
Marcol 172	mineral (ex Esso)	60.23	1.47056	0.859
Sirius M70	pure mineral (ex Silkolene)	24.38	1.46076	0.836
Sirius M180	mineral (ex Silkolene)	68.5	1.47171	0.860
Ryoto ER 290	Sugar ester (ex Mitsubishi Kagaku)	20x10 <sup>3</sup>	1.48354	0.968
Sirius M125	mineral (ex Silkolene)	42.524.38	1.46915	0.855
Sirius M350	mineral (ex Silkolene)	106.0	1.47451	0.866

(i) Softening evaluation method

Softening performance was evaluated by adding 0.1g of fabric softening compound to 1 litre of demineralised water at ambient temperature containing in a tergotometer. It should be noted that the level of actives was equal in the rinse liquor for the examples of the invention and the comparative examples. Three pieces of terry towelling (19cm x 19.5cm) were added to the tergotometer pot (The terry towelling was previously rinsed with 0.001% (w/w) sodium alkyl benzene sulphonate (ABS)) to simulate carryover of anionic detergent from the main wash.)

The cloths were treated for 5 minutes at 65 rpm, spin dried to remove excess liquor and line dried overnight. Softness was evaluated by a trained panel of four people ranked the cloths against set standards. A low number indicates a greater degree of softening.

(ii) Perfume delivery evaluation method

Perfume delivery was evaluated by rinsing three pieces of terry towelling (7.75 by 7.5"), per product in a similar manner to that previously described for softening evaluation above. Instead of being line-dried the cloths were immediately assessed for perfume intensity by a trained group of eighteen panellists who ranked each cloth on a scale of zero to five corresponding to descriptors ranging from no perfume to very strong perfume. Further assessments were made after five hours when the cloths were dry and again after twenty-four hours. The level of product was 0.1g/l active matter with a perfume level in the rinse liquor of 4.76mg/l.

Absorbency of fabrics was evaluated by treating fabric as described for the softening assessment. Strips of fabric were cut to 11 cm by 3 cm. The strips of treated fabric were held vertically and lowered into a dish containing a 0.02% solution of direct red 81 dye, so that ca. 0.5cm of the fabric was below the surface of the water. The height to which the liquid rose up the strip was measured at intervals of time for a total of one hour. The average height for each treatment was calculated. Higher values are indicative of better absorbency.

Examples 6,7 and I

Example 6 was prepared as for Examples 1 to 5.

Example 7 was prepared by adding Example 6 to hot water (60 °C to give an emulsion comprising 20% active. Example I a conventional softener dispersion, was prepared by stepwise addition of the components to hot water.

	Example 6	Example I
Sirius M70	53.23	-
Arquad 2HT	36.92	7.3
Coco20EO	5.91	-
Hardened Tallow fatty acid	-	0.4

(continued)

	Example 6	Example I
Perfume	3.9	0.3
Water	0.0	91.6

Softening and absorbency were evaluated as described above.

	Softening score	Absorbency Height (mm) in one hour
Example 6	3.38	126
Example 7	3.5	109
Example I	3.0	18

These results show that Examples 6 and 7 of the invention unlike comparative Example I have dissociated softening from absorbency.

The perfume performance, crease recovery, average recovery angle and ease of ironing for examples 6, F, G and H (as according to the present invention) and comparative Example I were tested as detailed below.

(i) Perfume Performance

Perfume Performance was evaluated as described above.

Table 3

	Wet Cloth	After 5 hours	After 24 hours
Example 6	2.58	2.39	1.74
Example I (comparative)	2.22	1.36	0.95
Example F	3.53	2.36	1.42
Example G	3.69	1.97	1.36
Example H	3.33	2.40	1.81

(ii) Crease Recovery (warp test)

Cotton poplin cloth was soaked in a solution of fabric softening compound (0.1g/l in demineralised water) at room temperature and then wrung using an Atlas Laboratory wringer. After line drying, the cloth was left at 20°C and 65% humidity for 24 hours. The cloth was then cut to 25mm to 50mm. The cloth was folded in half (short ends together) and placed on a plate. A 2kg load was placed on the crease of the cloth using a "Shirley Crease Recovery Tester (SDL 003A)". One half of the cloth was secured to the tester, the other half being left suspended in the air. The angle of the free end was measured relative to secured end after 2 minutes 30 seconds.

(iii) Average recovery angle (warp)

Example 6	73.0
Example 7	63.28
Example I	57.2

The greater the angle the better the crease recovery of the cloth.

(iv) Ease of ironing

From direct observation of the cloths it could be seen that the wrinkles fell out more easily from cloths treated with Example 6 than cloths treated with Example I. This made ironing of the cloth treated with Example 6 rinse conditioner

easier as the wrinkles needed little pressure from the iron to be removed. In this respect wrinkles fell out more easily from cloths treated with Example 6 than cloths treated with Example 7.

#### Examples 8 to 16

Table 4

COMPOSITION	Example 8 %	Example 9 %	Example 10 %	Example 11 %
Sirius M70	47.32	59.14	53.23	-
Arquad 2HT	36.92	36.92	36.92	36.92
Coco20EO	5.91	-	-	5.91
Hardened Tallow Fatty Acid	5.91	-	5.91	-
Perfume	3.94	3.94	3.94	3.92
Sirus M180	-	-	-	47.32

Table 5

COMPOSITION	EXAMPLES				
	%				
	12	13	14	15	16
Priolube 1446	65.03	56.11	-	-	-
Sirius M70	-	-	52.92	52.42	53.63
2HT	-	-	36.71	-	-
HEQ	30.64	39.69	-	37.27	-
Deedmac	-	-	-	-	35.91
Coco20EO	-	-	5.82	5.77	5.90
Tallow 15 EO	4.33	4.20	-	-	-
Perfume	-	-	4.55	4.54	4.54

HEQ is a mixture of 66.2% 1,2 bis[hardened tallowoyloxy]-3-trimethylammonium propane chloride, 11.03% tallow fatty acid and isopropyl alcohol

DEEDMAC is a mixture of di(hardened tallowoyloxyethyl)dimethyl ammonium chloride 83% quat, 2% tallow fatty acid and isopropyl alcohol.

All examples exhibited good softening of the fabrics and perfume delivery.

#### Examples 17 and 18; Cold grinding route of preparation

A further method of producing the compositions of the present invention is demonstrated by this example.

Example 17 had same composition as Example 15 (above) and Example 18 had the same composition as Example 16 except the oil was Estol 1545. In both cases the fabric softening compound (HEQ or Deedmac) was added to the oil (Sirius M70) and other ingredients followed by high-shear mixing at ambient temperature. The compositions produced were dispersions with relatively low viscosities. This method is referred to herein as the cold-grinding route.

The cold-grinding route produces products with typically lower viscosities than those produced by the melt route of the present invention. The cold-grinding route provides a particularly advantageous route for the preparation of products comprising high melting point fabric softening compounds.

Examples 15 and 16 as prepared by the melt route were substantially solid and difficult to disperse at ambient temperature. Examples 17 and 18 as prepared by the cold grinding route were soft solids/pastes.

The softening and perfume performance of the same compositions but produced by two routes of the invention were comparable.

Example 16 (melt) route and Example 18 (cold-grinding route) both exhibited excellent softening and perfume delivery/ longevity (see Table 6).

# EP 0 829 531 A1

Table 6

Example	Method of Preparation	Softening Score
16	melt	X
18	cold grinding	4.62
X could not be determined as could not be dispersed.		

## Examples 19 to 27

The following compositions were prepared according to the melt method of Example 1 but incorporating different solvents.

Table 7

	19	20	21	22	23	24
Arquad 2HT (excl. solvent)*	29.57	29.7	29.7	29.7	29.7	29.7
Sirius M70	53.23	53.47	0	0	0	0
Sirius M180	0	0	53.47	53.47	53.47	53.47
Perfume	3.94	2.97	2.97	2.97	2.97	2.97
Pristerine 4916	0	0.5	0.5	0.5	0.5	0.5
Genapol C200 (Coco 20EO)	5.91	0	0	0	0	0
Tergitol 15-S-7	0	5.94	5.94	5.94	5.94	5.94
IPA	7.35	4.52	3.72	1.86	0	0
Hexylene glycol	0	1.51	3.72	5.57	7.43	0
Buytl digol	0	0	0	0	0	7.43
Flash point /°C	36	39	48	65	>110	not measured

\* For all except 19 IPA was evaporated off raw material and pure solvents added to melt

Sirius M70 and Sirius M180 are mineral oils.  
Pristerine 4716 is a fatty acid (hardened Tallow).  
Genapol C200 and Tergitol 15-S-7, are nonionic surfactants.

Table 8

Examples	25	26	27
	Wt. %	Wt. %	Wt. %
DEEDMAC (excluding solvent)	27.95	27.6	25.62
IPA (from DEEDMAC raw material)	0	4.87	4.52
Propylene glycol (from DEEDMAC raw material)	4.93	0	0
Estol 1545 ester oil	0	0	44.2
Sirius M70 mineral oil	54.02	47.62	0
Perfume	3.73	3.63	3.38
Pristerine 4916 fatty acid	0.93	0.9	0.83
Genapol C200	1.4	0	0
Genapol C065	0	5.39	0
Genapol C050	0	0	5.00
Hexylene glycol	7.04	9.99	16.45

Table 8 (continued)

Examples	25	26	27
Flash point /°C	not measured	63	not measured

The compositions exhibited good viscosity appearance and where measured, acceptable flash points.

#### Examples 28 to 37

Compositions comprising a co-emulsifier or a mixture of co-emulsifier were prepared by the melt process as according to Example see Table 9 and 10. The dispersion characteristic of the compositions in water was assessed as given below.

Table 10

Examples	28	29	30	31	32
	Wt. %	Wt. %	Wt. %	Wt. %	Wt. %
Arquad 2HT with solvent removed	29.7	29.7	29.7	29.7	29.7
Sirius M180	53.47	53.47	53.47	53.47	53.47
Perfume	2.97	2.97	2.97	2.97	2.97
Pristerine 4916 fatty acid	0.5	0.5	0.5	0.5	0.5
Hexylene glycol	7.43	7.43	7.43	7.43	7.43
Genapol C200 (Coco 20EO nonionic	5.94	4.46	2.97	1.49	0
Tergitol 15-S-7	0	1.49	2.97	4.46	5.94

Table 10

Examples	33	34	35	36	37
	Wt. %	Wt. %	Wt. %	Wt. %	Wt. %
Arquad 2HT with solvent removed	29.7	29.7	29.7	29.7	29.7
Sirius M180 mineral oil	53.47	53.47	53.47	53.47	53.47
Perfume	2.97	2.97	2.97	2.97	2.97
Pristerine 4916 fatty acid	0.5	0.5	0.5	0.5	0.5
Hexylene glycol	7.43	7.43	7.43	7.43	7.43
Synperonic A20	2.97	4.46	1.49	5.94	0
Synperonic A7	2.97	1.49	4.46	0	0
Genapol C050 (Coco 5EO nonionic)	0	0	0	0	5.94

#### Dispersion Test

Dispersion was assessed by turbidity. Equal weights of the compositions were added to stirred water at 10°C and the change in turbidity measured with time. A turbidity curve was achieved which initially rose as dispersion took place, then reached a plateau when dispersion was complete. To assess the rate of dispersion the turbidity after 12s compared to the plateau turbidity was measured expressed as % dispersion after 12s.

Table 11

Composition	% dispersion after 12s	
28	69	Synperonic A20
29	75	Synperonic A20-Synperonic A7 3:1
30	85	Synperonic A20: Synperonic A7 1:1

Table 11 (continued)

Composition	% dispersion after 12s	
31	88	Synperonic A20: Synperonic A7 1:3
32	76	Coco20EO
33	83	Coco20EO : Tergitol 15-S-7 3:1
34	94	Coco20EO : Tergitol 15-S-7 1:1
35	86	Coco20EO : Tergitol 15-S-7 1:3
36	94	Tergitol 15-S-7
37	81	Coco 5EO

Genapol C200 Ex. Hoechst, primary alcohol ethoxylate with coco (mainly C<sub>12-14</sub> carbon chain and average 20 ethoxylate headgroup, HLB 16.4 (calculated).

Genapol C050 As above, but average 5 ethoxylate headgroup, HLB 11 (calculated).

Synperonic A20 Ex. Shell, alcohol ethoxylate with mainly C<sub>13</sub> carbon chain with some branching, and average 20 ethoxylate headgroup, HLB 16.2 (calculated)

Synperonic A7 As above, but average 7 ethoxylate group, HLB 12.8 (calculated)

Tergitol 15-S-7 Ex. Union Carbide, secondary alcohol ethoxylate with mainly C<sub>12</sub>-C<sub>14</sub> carbon chain and average 7 ethoxylate headgroup, HLB 12.7 (calculated)

Genapol, Synperonic and Tergitol are trademarks.

This demonstrates that although the dispersion of the compositions with high HLB surfactants alone is reasonably rapid, it becomes significantly faster when low HLB surfactant is added. For some systems just 25% of the surfactant being low HLB will give large improvement, but for others at least 50% low HLB is preferred.

#### Example 38

Comparisons comprising blends of oils were prepared as below. The compositions of examples 38 to 42 are as given for Example 9. In each case a mineral oil was blended with petroleum jelly in various ratios. In each case Sirius M70 mineral oil was used. Examples 41 and 42 act as comparative examples.

Table 12

Example	Mineral oil: Petroleum Jelly	Softness Score	Perfume Wet	Perfume 24 hours
38	25:75	5.25	3.33	1.69
39	50:50	4.37	3.57	1.88
40	75:25	-	3.67	1.98
41	0:100	4.87	3.52	1.64
42	100:0	4.62	3.50	2.42

The above demonstrates that the softness results and perfume longevity are maintained across a range of oil blend ratios.

#### Claims

1. A conditioning concentrate composition comprising a cationic fabric softening compound and an oil in which the cationic fabric softening compound is suspended in the oil.

2. A conditioning concentrate according to claim 1 which further comprises a crystal growth inhibitor.
3. A conditioning concentrate according to either of claims 1 or 2 in which the oil is an ester oil, mineral oil or sugar ester oil.
- 5 4. A conditioning concentrate according to claim 3 in which the ester oil or mineral oil has a viscosity from 2 cP (mPa.s) to 150 cP (mPa.s) at 25°C or the ester oil has a viscosity above 5000 cP.
- 10 5. A conditioning concentrate according to any preceding claim in which the oil is a saturated oil.
6. A conditioning concentrate according to any one of claims 2-5 in which the crystal growth inhibitor is a organic acid with an alkyl chain length of C<sub>18</sub> or less, or a C<sub>10</sub>-C<sub>22</sub> alkyl chain nonionic ethoxylate having from 10 to 30 ethoxylate groups.
- 15 7. A conditioning concentrate according to any preceding claim in which the fabric softening compound is a quaternary ammonium material comprising a polar head group and two alkyl or alkenyl chains having an average chain length greater than C<sub>14</sub>.
- 20 8. A conditioning concentrate according to any preceding claim in which the ratio of cationic softening compound to oil is from 1:5 to 1:1.
9. A conditioning concentrate according to any preceding claim in which the concentrate has a level of water of 5 wt% or less.
- 25 10. A conditioning concentrate according to any preceding claim in which the concentrate comprises a co-emulsifier.
11. A conditioning concentrate according to claim 10 in which the co-emulsifier is a mixture of a surfactant having an HLP of 14 or less and a surfactant having an HLB of 15.5 or greater.
- 30 12. A conditioning concentrate according to any preceding claim in which the composition comprises an organic solvent in an amount of less than 25% by weight.
13. A conditioning concentrate according to any preceding claim in which the softening compound and the oil are heated together to form a melt.
- 35 14. A conditioning concentrate according to claim 13 in which the crystal growth inhibitor forms part of the melt.
15. A conditioning concentrate according to any preceding claim in which the softening compound and the oil are mixed together at high shear at ambient temperature.
- 40 16. A conditioning concentrate according to any preceding claim in which the cationic fabric softening compound is suspended as a solid crystalline phase.
17. Use of a composition according to any preceding claim to reduce creasing of fabric, and or to improve water absorbency of a fabric.
- 45 18. A process for perfuming and softening laundry in which the composition of any one of claims 1 to 12 is added directly to the rinse liquor.



European Patent  
Office

# EUROPEAN SEARCH REPORT

Application Number  
EP 97 30 6076

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.CI.6)
D,X	GB 2 007 734 A (CARGO FLEET CHEMICAL CO) 23 May 1979 * entire document *	1-18	C11D3/00 C11D1/62 C11D3/18 C11D3/20 C11D3/43 C11D3/50 C11D1/72
X	DE 42 43 862 A (HUELS CHEMISCHE WERKE AG) 30 June 1994 * claims 1-4; examples 1-6 *	1-18	
A	WO 94 19439 A (UNILEVER PLC ; UNILEVER NV (NL)) 1 September 1994 * entire document *	1-18	
A	WO 95 22594 A (UNILEVER PLC ; UNILEVER NV (NL)) 24 August 1995 * examples 1-10 *	1-18	
			TECHNICAL FIELDS SEARCHED (Int.CI.6)
			C11D
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 11 December 1997	Examiner Ainscow, J
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons &amp; : member of the same patent family, corresponding document</p>			

EP0 FORM 1503 03/92 (P4/C01)