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(54) Title: IMPROVEMENTS RELATING TO FABRIC CONDITIONERS

(57) **Abstract**: A process for preparing an aqueous liquid fabric conditioning composition comprising a water insoluble ester-linked quaternary ammonium softening active, and a water soluble quaternary ammonium surfactant, wherein the process comprises the step of dispersing the water soluble quaternary ammonium surfactant in water before the addition of the water insoluble ester-linked quaternary ammonium softening active to the water, leads to improved storage stability at elevated temperatures.

IMPROVEMENTS RELATING TO FABRIC CONDITIONERS

Technical Field

The present invention concerns a process for the preparation of opaque fabric conditioner compositions comprising an encapsulated perfume, a water insoluble ester-linked quaternary ammonium softening active and a water soluble quaternary ammonium surfactant.

Background of the Invention

In hot climates, significant thickening of liquid laundry compositions such as fabric conditioners can occur during storage. The performance of these products can consequently be negatively affected with a resultant reduction in the desired benefit to the consumer. Furthermore, the addition of encapsulated perfumes to fabric conditioners is known to have a further detrimental effect on the stability of fabric conditioners.

US5861371 describes the preparation of aqueous fabric conditioners that are preferably water-clear in appearance, which comprise up to 30 wt % of a water insoluble quaternary ammonium compound, up to 50 wt % of a water soluble quaternary ammonium compound, a water soluble acid and an emulsifier comprising an ethylene oxide adduct of a hydroxyl functional fatty compound having an HLB value of 12 to 19.

WO93/23510 discloses concentrated fabric softener compositions comprising an ester linked fabric softener active and an optional water soluble quaternary ammonium compound. A lab scale preparation method is exemplified, whereby all components are mixed together in a sealed jar. Aging of the resultant compositions causes an increase in viscosity in as little as only 1 day at ambient temperature.

The use of a water soluble quaternary ammonium cationic surfactant to improve the stability of fabric conditioners comprising non-ester quaternary ammonium conditioning agents is known from EP1922397A1. This patent discloses that an order of addition wherein a slurry of encapsulated perfume is added to charge water and water soluble cationic surfactant is added after the addition of water insoluble fabric softening agent and any free perfume at a temperature below the phase transition temperature of the composition, results in excellent long term stability.

We have, however, found that this method of preparation results in catastrophic stability failure in fabric conditioner compositions comprising an ester-containing quaternary ammonium softening active, for example a triethanoloamine based quaternary compound. There remains a need for ways of improving stability of such fabric conditioner compositions, which is effective at elevated temperatures.

The present invention uses a water soluble quaternary ammonium surfactant to increase the storage stability of a laundry composition, comprising an ester-linked quaternary ammonium softening active, at elevated temperatures. We have surprisingly found that the incorporation of this material into the product using a specific process route, whereby the water soluble quaternary ammonium surfactant is added to the water phase before the addition of the molten active phase, is critical to achieving improved viscostability of the end product, and confers stable viscosity for at least 12 weeks at elevated temperatures.

Statement of the Invention

In a first aspect of the invention there is provided a process for preparing an opaque, aqueous liquid fabric conditioning composition comprising:

(a) a water insoluble ester-linked quaternary ammonium softening active, which, in pure form as a strong acid salt, has a solubility in distilled water at pH 2.5 and 20°C of less than 1g/l, and

(b) a water soluble quaternary ammonium surfactant, which is a cationic surfactant having a solubility in distilled water at pH 2.5 and 20°C of greater than 1g/l,

(c) an encapsulated perfume,

wherein the process comprises the step of dispersing the water soluble quaternary ammonium surfactant in water before the addition of the water insoluble ester-linked quaternary ammonium softening active to the water.

In a second aspect of the invention, there is provided a composition obtained from the process of the first aspect of the invention.

Detailed Description of the Invention

The Process

In the process of the invention, the water soluble quaternary ammonium surfactant is added to a water phase, before addition of a melt phase comprising molten water insoluble ester-linked quaternary ammonium softening active. The water phase may also contain minor components, for example, preservatives and antifoam.

Encapsulated perfume may be added to the water phase. Non-confined perfume oil is preferably added in the conventional way, after the active and water phases have been combined and cooled.

A preferred process of the invention comprises the steps of:-

1) mix the water soluble quaternary ammonium surfactant and perfume encaps [along with optional ingredients, for example, polymer and minors], with heated water to form a water phase;
2) melt the water insoluble ester-linked quaternary ammonium softening active [and optional non-ionic surfactant] to form a melt;
3) combine the water phase and the melt with agitation;
4) add dye, pearlescer and electrolyte if required;
5) allow the resulting mixture to cool; and
6) add any non-confined perfume oil and optional antifoam to the cooled mixture.

Preferably, the optional ingredients (polymer, minors, perfume encaps, etc) are added to the heated water before the addition of the water soluble quaternary ammonium surfactant.

Viscosity of the compositions may suitably be measured on a cup and bob viscometer; the viscosity being continuously measured under shear at $106s^{-1}$ for 60 seconds, at 25°C.

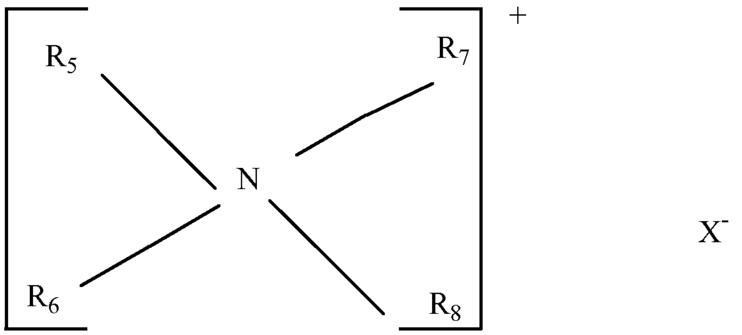
Preferably the compositions made by the process of the invention have a viscosity in the range of from 50 to 150 mPas-1, more preferably from 70 to 110 mPas-1.

The Water-Soluble Quaternary Ammonium Surfactant

The water-soluble quaternary ammonium surfactant is a cationic surfactant having a solubility in distilled water at pH 2.5 and 20°C of greater than 1g/l. The solubility of the water-soluble quaternary ammonium surfactant is defined with reference to the pure material in the form of a strong acid salt (e.g. chloride), and the soluble fraction of the surfactant is taken to be that material which cannot be separated from water by centrifugal action and which passes a 100nm Nuclepore filter (registered trade mark).

Preferred water-soluble quaternary ammonium surfactants are mono-C_8-C_{24} alkyl or alkenyl ammonium salts, imidazolinium salts, pyridinium salts and mixtures thereof.

Suitable water-soluble mono-ammonium surfactant compounds have the general formula:



wherein:
R_5 represents a C_8-C_{24} alkyl or alkenyl group,
R_6 represents hydrogen, a C_1-C_{12} alkyl, alkenyl or hydroxyalkyl group, an aryl group, a

C_{1-6} alkylaryl group, or a poly(ethylene oxide) group having from 2 to 20 ethylene oxide units,
R_7 and R_8 individually represent hydrogen, a C_1-C_4 alkyl, alkenyl or hydroxyalkyl group or a poly(ethylene oxide) group having from 2 to 20 ethylene oxide units and
X is the salt counter-anion, preferably selected from halide, methyl sulfate and ethyl sulfate radicals.

Preferred materials of this general type include the tallow trimethyl ammonium salts, cetyl trimethyl ammonium salts, myristyl trimethyl ammonium salts, coconut alkyl trimethyl ammonium salts, cetyl dimethyl ammonium salts, myristyl dimethyl ammonium salts, benzalkonium chloride, coconut alkyl dimethyl ammonium salts, oleyl methyl ammonium salts, palmityl methyl ammonium salts, myristyl methyl ammonium salts, lauryl methyl ammonium salts, dodecyl dimethyl hydroxyethyl ammonium salts, dodecyl dimethyl hydroxypropyl ammonium salts, myristyl dimethyl hydroxyethyl ammonium salts, dodecyl dimethyl dioxyethylenyl ammonium salts, myristyl benzyl hydroxyethyl methyl ammonium salts, coconut alkyl benzyl hydroxyethylmethyl ammonium salts, dodecyl dihydroxyethyl methyl ammonium salts, cetyl dihydroxyethyl methyl ammonium salts, stearyl dihydroxyethyl methyl ammonium salts and a mixture of octyldecyldimethyl ammonium chloride, didecyldimethyl ammonium chloride and dioctyldimethyl ammonium chloride.

The most preferred compounds are selected from cetyl trimethyl ammonium chloride, benzalkonium chloride and a mixture of octyldecyldimethyl ammonium chloride, didecyldimethyl ammonium chloride and dioctyldimethyl ammonium chloride, most preferably benzalkonium chloride.

The Water Insoluble Ester-linked Quaternary Ammonium Softening Active

The water insoluble ester-linked quaternary ammonium softening active is a fabric-substantive quaternary ammonium compound which, in pure form as a strong acid salt (e.g. chloride), has a solubility in distilled water at pH 2.5 and 20°C of less than 1g/l, preferably less than 0.1g/l more preferably less than 0.01 g/l or can be a mixture of such compounds. In this context, the soluble fraction of the surfactant is taken to be that material which cannot be separated from water by centrifugal action and which passes a 100nm Nuclepore filter (registered trade mark).

Preferred materials are compounds having two $-C_{12}$-C_{24} alkyl or alkenyl groups, or a quaternary ammonium compound comprising a single chain with an average chain length equal to or greater than C_{20}.

The water insoluble ester-linked quaternary ammonium softening active for use in the process and compositions of the invention is an ester-linked quaternary ammonium compound.

Preferred quaternary ammonium compounds for use in the process of the invention have unsaturated chains, i.e. are the so-called "soft" quats. Such compounds are typically derived from fatty acyl or fatty acid feed stock having an Iodine Value of from 20 to 140, preferably from 20 to 60, more preferably from 20 to 50, most preferably from 25 to 45. The unsaturated chains come from the unsaturated fatty feed stock.

If there is a mixture of quaternary ammonium materials present in the composition, the iodine value, referred to above, represents the mean iodine value of the parent fatty acyl compounds or fatty acids of all of the quaternary ammonium materials present. Likewise, if there is any saturated quaternary ammonium quaternary ammonium materials present in the composition, the iodine value, referred to above, represents the mean iodine value of the parent fatty acyl compounds or fatty acids of all of the quaternary ammonium materials present.

Iodine value is defined as the number of grams of iodine absorbed per 100 g of test material. NMR spectroscopy is a suitable technique for determining the iodine value of the softening agents of the present invention, using the method described in *Anal. Chem.*, 34, 1136 (1962) by Johnson and Shoolery and in EP 593,542 (Unilever, 1993).

The quaternary ammonium compound is preferably present in the compositions of the invention at a level of from 2 % to 55 %, preferably from 3 % to 50 %, more preferably from 4 % to 40 % even more preferably from 5 % to 25 %, still more preferably from 7 % to 14 %, by weight of the total composition, for example from 8 to 12 % by weight. The compositions of the invention are preferably concentrated fabric conditioners.

Particularly preferred materials are water insoluble ester-linked triethanolammonium (TEA) quaternary ammonium compounds comprising a mixture of mono-, di- and tri-ester linked components.

Typically, such TEA-based fabric softening compounds comprise a mixture of mono, di- and tri-ester forms of the compound. Typically the di-ester linked component comprises no more than 70 % by weight of the fabric softening compound, preferably no more than 60 %, e.g. no more than 55 %, or even no more than 45 % of the fabric softening compound and at least 10 % of the monoester linked component.

A first group of quaternary ammonium compounds (QACs) suitable for use in the present invention is represented by formula (I):

$$[(CH_2)_n(TR)]_m\text{-}(R^1).N^+\text{-}[(CH_2)_n(OH)]_{3-m}\ X^- \qquad (I)$$

wherein each R is independently selected from a C_{5-35} alkyl or alkenyl group; R^1 represents a C_{1-4} alkyl, C_{2-4} alkenyl or a C_{1-4} hydroxyalkyl group; T is generally O-CO. (i.e. an ester group bound to R *via* its carbon atom), but may alternatively be CO.O (i.e. an ester group bound to R *via* its oxygen atom); n is a number selected from 1 to 4; m is a number selected from 1, 2, or 3; and X^- is an anionic counter-ion, such as a halide or alkyl sulphate, e.g. chloride or methylsulphate. Di-esters variants of formula I (i.e. m = 2) are preferred and typically have mono- and tri-ester analogues associated with them. Such materials are particularly suitable for use in the present invention.

Most preferably, n = 2 and T = O-CO, to form a triethanolamine quaternary ammonium compound.

Especially preferred agents are preparations which are rich in the di-esters of triethanolammonium methylsulphate, otherwise referred to as “TEA ester quats”.

Commercial examples include Stepantex™ UL85, ex Stepan, Prapagen™ TQL, *ex* Clariant, and Tetranyl™ AHT-1, *ex* Kao, (both di-[hardened tallow ester] of triethanolammonium methylsulphate), AT-1 (di-[tallow ester] of triethanolammonium methylsulphate), and L5/90 (di-[palm ester] of triethanolammonium methylsulphate), both *ex* Kao, and Rewoquat™ WE15 (a di-ester of triethanolammonium methylsulphate having fatty acyl residues deriving from C_{10}-C_{20} and C_{16}-C_{18} unsaturated fatty acids), *ex* Degussa.

Also, soft quaternary ammonium actives such as Stepantex VK90, Stepantex VT90, SP88 (ex-Stepan), Prapagen TQ (ex-Clariant), Dehyquart AU-57 (ex-Cognis), Rewoquat WE18 (ex-Degussa) and Tetranyl L190 P, Tetranyl L190 SP and Tetranyl L190 S (all ex-Kao) are suitable.

A second group of quaternary ammonium compounds suitable for use in the invention is represented by formula (II):

$(R^1)_3N^+{-}(CH_2)_n\text{-}CH.(CH_2TR^2)\text{-}TR^2\ X^-$ (II)

wherein each R^1 group is independently selected from C_{1-4} alkyl, hydroxyalkyl or C_{2-4} alkenyl groups; and wherein each R^2 group is independently selected from C_{8-28} alkyl or alkenyl groups; and wherein n, T, and X^- are as defined above. Preferred materials of this second group include 1,2 *bis*[tallowoyloxy]-3-trimethylammonium propane chloride, 1,2 and 1,2-*bis*[oleoyloxy]-3-trimethylammonium propane chloride. Such materials are described in US 4,137,180 (Lever Brothers). Preferably, these materials also comprise an amount of the corresponding mono-ester.

A third group of quaternary ammonium compounds suitable for use in the invention is represented by formula (III):

$(R^1)_2\text{-}N^+\text{-}[(CH_2)_n\text{-}T\text{-}R^2]_2\ X^-$ (III)

wherein each R^1 group is independently selected from C_{1-4} alkyl, or C_{2-4} alkenyl groups; and wherein each R^2 group is independently selected from C_{8-28} alkyl or alkenyl groups; and n, T, and X^- are as defined above. Preferred materials of this third group include bis(2-tallowoyloxyethyl)dimethyl ammonium chloride.

The Encapsulated Perfume

The compositions derived from the process of the present invention comprise comprise one or more encapsulated perfumes. Non-encapsulated (also called non-confined, or free perfume) perfume may also be present. The total amount of encapsulated and non-confined perfume present is preferably an amount of from

0.01 to 10 % by weight, more preferably from 0.05 to 5 % by weight, even more preferably from 0.1 to 4.0 %, most preferably from 0.5 to 3.0 % by weight, based on the total weight of the composition. The amount of encaps present is preferably from 0.01 to 0.9 %, more preferably from 0.05 to 0.7 and most preferably from 0.1 to 0.5 % by weight of the total composition.

Encapsulated Perfume

The fabric conditioners of the present invention comprise an encapsulated perfume. The encapsulated perfumed is preferably present in an amount of from 0.1 to 5 wt %, by weight of the total composition.

The encapsulated perfume is preferably in the form of a slurry having a viscosity of from greater than water to 1000 cps at 21 s^{-1} and 25 °C.

The perfume loading of the encaps, that is to say the amount of the total encap weight that is perfume, is preferably from 20 to 40 wt %, more preferably from 28 to 32 wt %, by total weight of the encaps.

The encaps (or "capsules") for use in the process of the present invention comprise a shell. The shell is preferably comprised of materials including aminoplasts, proteins, polyurethanes, polysaccharides, gums, celluloses, and any other encapsulating material which may be used effectively in the present invention, such as polymethylmethacrylate. Preferred encapsulating polymers include those formed from melamine formaldehyde or urea formaldehyde condensates, as well as similar types of aminoplasts. Most preferably the shell comprises melamine formaldehyde.

Additionally, microcapsules made via the simple or complex coacervation of gelatin are also preferred for use with the coating. Microcapsules having shell walls comprised of polyurethane, polyamide, polyolefin, polysaccaharide, protein, silicone, lipid, modified cellulose, gums, polyacrylate, polystyrene, and polyesters or combinations of these materials are also possible.

A representative process used for aminoplast encapsulation is disclosed in U.S. Patent No. 3,516,941 though it is recognized that many variations with regard to materials and process steps are possible. A representative process used for gelatin encapsulation is disclosed in U.S. Patent No, 2,800,457 though it is recognized that many variations with regard to materials and process steps are possible. Both of these processes are discussed in the context of fragrance encapsulation for use in consumer products in U.S. Patent Nos. 4,145,184 and US 5,112,688 respectively.

Encapsulation can provide pore vacancies or interstitial openings depending on the encapsulation techniques employed.

Fragrance capsules known in the art and suitable for use in the present invention comprise a wall or shell comprising a three-dimensional cross-linked network of an aminoplast resin, more specifically a substituted or un-substituted acrylic acid polymer or co-polymer cross-linked with a urea-formaldehyde pre-condensate or a melamine-formaldehyde pre-condensate.

Microcapsule formation using mechanisms similar to the foregoing mechanism, using (i) melamine-formaldehyde or urea-formaldehyde pre-condensates and (ii) polymers containing substituted vinyl monomeric units having proton-donating functional group moieties (e.g. sulfonic acid groups or carboxylic acid anhydride groups) bonded thereto is disclosed in U.S. Patent 4,406,816 (2-acrylamido-2-methyl-propane sulfonic acid groups), UK published Patent Application GB 2,062,570 A (styrene sulfonic acid groups) and UK published Patent Application GB 2,006,709 A (carboxylic acid anhydride groups).

Particle size and average diameter of the capsules can vary from about 10 nanometers to about 1000 microns, preferably from about 50 nanometers to about 100 microns, more preferably from about 2 to about 40 microns, even more preferably from about 3 to 30 microns. A particularly preferred range is from about 5 to 10 microns, for example 6 to 7 microns. The capsule distribution can be narrow, broad or multimodal. Multimodal distributions may be composed of different types of capsule chemistries.

Perfume Composition

Useful components of the perfume include materials of both natural and synthetic origin. They include single compounds and mixtures. Specific examples of such components may be found in the current literature, e.g., in Fenaroli's Handbook of Flavor Ingredients, 1975, CRC Press; Synthetic Food Adjuncts, 1947 by M. B. Jacobs, edited by Van Nostrand; or Perfume and Flavor Chemicals by S. Arctander 1969, Montclair, N.J. (USA). These substances are well known to the person skilled in the art of perfuming, flavouring, and/or aromatizing consumer products, i.e., of imparting an odour and/or a flavour or taste to a consumer product traditionally perfumed or flavoured, or of modifying the odour and/or taste of said consumer product.

By perfume in this context is not only meant a fully formulated product fragrance, but also selected components of that fragrance, particularly those which are prone to loss, such as the so-called ‘top notes’.

Top notes are defined by Poucher (Journal of the Society of Cosmetic Chemists 6(2):80 [1955]). Examples of well known top-notes include citrus oils, linalool, linalyl acetate, lavender, dihydromyrcenol, rose oxide and cis-3-hexanol. Top notes typically comprise 15-25%wt of a perfume composition and in those embodiments of the invention which contain an increased level of top-notes it is envisaged at that least 20%wt would be present within the encapsulate.

It is advantageous to encapsulate perfume components which have a low Clog P (i.e. those which will be partitioned into water), preferably with a Clog P of less than 3.0. These materials, of relatively low boiling point and relatively low Clog P have been called the “delayed blooming” perfume ingredients and include the following materials:

Allyl Caproate, Amyl Acetate, Amyl Propionate, Anisic Aldehyde, Anisole, Benzaldehyde, Benzyl Acetate, Benzyl Acetone, Benzyl Alcohol, Benzyl Formate, Benzyl Iso Valerate, Benzyl Propionate, Beta Gamma Hexenol, Camphor Gum, Laevo-Carvone, d-Carvone, Cinnamic Alcohol, Cinamyl Formate, Cis-Jasmone, cis-3-Hexenyl Acetate, Cuminic Alcohol, Cyclal C, Dimethyl Benzyl Carbinol, Dimethyl Benzyl Carbinol Acetate, Ethyl Acetate, Ethyl Aceto Acetate, Ethyl Amyl Ketone, Ethyl Benzoate, Ethyl Butyrate, Ethyl Hexyl Ketone, Ethyl Phenyl Acetate, Eucalyptol, Eugenol, Fenchyl Acetate, Flor Acetate (tricyclo Decenyl Acetate), Frutene (tricyclco Decenyl Propionate), Geraniol, Hexenol, Hexenyl Acetate, Hexyl Acetate, Hexyl Formate, Hydratropic Alcohol, Hydroxycitronellal, Indone, Isoamyl Alcohol, Iso Menthone, Isopulegyl Acetate, Isoquinolone, Ligustral, Linalool, Linalool Oxide, Linalyl Formate, Menthone, Menthyl Acetphenone, Methyl Amyl Ketone, Methyl Anthranilate, Methyl Benzoate, Methyl Benyl Acetate, Methyl Eugenol, Methyl Heptenone, Methyl Heptine Carbonate, Methyl Heptyl Ketone, Methyl Hexyl Ketone, Methyl Phenyl Carbinyl Acetate, Methyl Salicylate, Methyl-N-Methyl Anthranilate, Nerol, Octalactone, Octyl Alcohol, p-Cresol, p-Cresol Methyl Ether, p-Methoxy Acetophenone, p-Methyl Acetophenone, Phenoxy Ethanol, Phenyl Acetaldehyde, Phenyl Ethyl Acetate, Phenyl Ethyl Alcohol, Phenyl Ethyl Dimethyl Carbinol, Prenyl Acetate, Propyl Bornate, Pulegone, Rose Oxide, Safrole, 4-Terpinenol, Alpha-Terpinenol, and /or Viridine.

Suitable non-encapsulated perfume ingredients include those hydrophobic perfume components with a ClogP above 3. As used herein, the term "ClogP" means the logarithm to base 10 of the octanol/water partition coefficient (P). The octanol/water partition coefficient of a PRM is the ratio between its equilibrium concentrations in octanol and water. Given that this measure is a ratio of the equilibrium concentration of a PRM in a non-polar solvent (octanol) with its concentration in a polar solvent (water), ClogP is also a measure of the hydrophobicity of a material--the higher the ClogP value, the more hydrophobic the material. ClogP values can be readily calculated from a program called "CLOGP" which is available from Daylight Chemical Information Systems Inc., Irvine Calif., USA. Octanol/water partition coefficients are described in more detail in U.S. Pat. No. 5,578,563.

Perfume components with a ClogP above 3 comprise: Iso E super, citronellol, Ethyl cinnamate, Bangalol, 2,4,6-Trimethylbenzaldehyde, Hexyl cinnamic aldehyde, 2,6-Dimethyl-2-heptanol, Diisobutylcarbinol, Ethyl salicylate, Phenethyl isobutyrate, Ethyl hexyl ketone, Propyl amyl ketone, Dibutyl ketone, Heptyl methyl ketone, 4,5-Dihydrotoluene, Caprylic aldehyde, Citral, Geranial, Isopropyl benzoate, Cyclohexanepropionic acid, Campholene aldehyde, Caprylic acid, Caprylic alcohol, Cuminaldehyde, 1-Ethyl-4-nitrobenzene, Heptyl formate, 4-Isopropylphenol, 2-Isopropylphenol, 3-Isopropylphenol, Allyl disulfide, 4-Methyl-1-phenyl-2-pentanone, 2-Propylfuran, Allyl caproate, Styrene, Isoeugenyl methyl ether, Indonaphthene, Diethyl suberate, L-Menthone, Menthone racemic, p-Cresyl isobutyrate, Butyl butyrate, Ethyl hexanoate, Propyl valerate, n-Pentyl propanoate, Hexyl acetate, Methyl heptanoate, trans-3,3,5-Trimethylcyclohexanol, 3,3,5-Trimethylcyclohexanol, Ethyl p-anisate, 2-Ethyl-1-hexanol, Benzyl isobutyrate, 2,5-Dimethylthiophene, Isobutyl 2-butenoate, Caprylnitrile, gamma-Nonalactone, Nerol, trans-Geraniol, 1-Vinylheptanol, Eucalyptol, 4-Terpinenol, Dihydrocarveol, Ethyl 2-methoxybenzoate, Ethyl cyclohexanecarboxylate, 2-Ethylhexanal, Ethyl amyl carbinol, 2-Octanol, 2-Octanol, Ethyl methylphenylglycidate, Diisobutyl ketone, Coumarone, Propyl isovalerate, Isobutyl butanoate, Isopentyl propanoate, 2-Ethylbutyl acetate, 6-Methyl-tetrahydroquinoline, Eugenyl methyl ether, Ethyl dihydrocinnamate, 3,5-Dimethoxytoluene, Toluene, Ethyl benzoate, n-Butyrophenone, alpha-Terpineol, Methyl 2-methylbenzoate, Methyl 4-methylbenzoate, Methyl 3, methylbenzoate, sec.Butyl n-butyrate, 1,4-Cineole, Fenchyl alcohol, Pinanol, cis-2-Pinanol, 2,4, Dimethylacetophenone, Isoeugenol, Safrole, Methyl 2-octynoate, o-Methylanisole, p-Cresyl methyl ether, Ethyl anthranilate, Linalool, Phenyl butyrate, Ethylene glycol dibutyrate, Diethyl phthalate, Phenyl mercaptan, Cumic alcohol, m-Toluquinoline, 6-Methylquinoline, Lepidine, 2-Ethylbenzaldehyde, 4-Ethylbenzaldehyde, o-Ethylphenol, p-Ethylphenol, m-Ethylphenol, (+)-Pulegone, 2,4-Dimethylbenzaldehyde, Isoxylaldehyde, Ethyl sorbate, Benzyl propionate, 1,3-Dimethylbutyl acetate, Isobutyl isobutanoate, 2,6-Xylenol, 2,4-Xylenol, 2,5-Xylenol, 3,5-Xylenol, Methyl cinnamate, Hexyl methyl ether, Benzyl ethyl ether, Methyl salicylate, Butyl propyl ketone, Ethyl amyl ketone, Hexyl methyl ketone, 2,3-Xylenol, 3,4, Xylenol, Cyclopentadenanolide and Phenyl ethyl 2 phenylacetate 2.

It is commonplace for a plurality of perfume components to be present in a formulation.

Another group of perfumes with which the present invention can be applied are the so-called ‘aromatherapy’ materials. These include many components also used in perfumery, including components of essential oils such as Clary Sage, Eucalyptus, Geranium, Lavender, Mace Extract, Neroli, Nutmeg, Spearmint, Sweet Violet Leaf and Valerian.

Optional Ingredients

Co-softeners and Fatty Complexing Agents

Co-softeners may be used. When employed, they are typically present at from 0.1 to 20% and particularly at from 0.5 to 10%, based on the total weight of the composition. Preferred co-softeners include fatty esters, and fatty N-oxides. Fatty esters that may be employed include fatty monoesters, such as glycerol monostearate, fatty sugar esters, such as those disclosed WO 01/46361 (Unilever).

The compositions of the present invention will preferably comprise a fatty complexing agent. Especially suitable fatty complexing agents include fatty alcohols and fatty acids. Of these, fatty alcohols are most preferred.

Without being bound by theory it is believed that the fatty complexing material improves the viscosity profile of the composition by complexing with mono-ester component of the fabric conditioner material thereby providing a composition which has relatively higher levels of di-ester and tri-ester linked components. The di-ester and tri-ester linked components are more stable and do not affect initial viscosity as detrimentally as the mono-ester component.

It is also believed that the higher levels of mono-ester linked component present in compositions comprising quaternary ammonium materials based on TEA may destabilise the composition through depletion flocculation. By using the fatty complexing material to complex with the mono-ester linked component, depletion flocculation is significantly reduced.

In other words, the fatty complexing agent at the increased levels, as required by the present invention, "neutralises" the mono-ester linked component of the quaternary ammonium material. This in situ di-ester generation from mono-ester and fatty alcohol also improves the softening of the composition.

Preferred fatty acids include hardened tallow fatty acid (available under the trade name Pristerene™, ex Uniqema). Preferred fatty alcohols include hardened tallow alcohol (available under the trade names Stenol™ and Hydrenol™, ex Cognis and Laurex™ CS, ex Albright and Wilson).

The fatty complexing agent is preferably present in an amount greater than 0.3 to 5% by weight based on the total weight of the composition. More preferably, the fatty component is present in an amount of from 0.4 to 4%. The weight ratio of the mono-ester component of the quaternary ammonium fabric softening material to the fatty complexing agent is preferably from 5:1 to 1:5, more preferably 4:1 to 1:4, most preferably 3:1 to 1:3, e.g. 2:1 to 1:2.

Nonionic Surfactant Actives

The compositions may further comprise a nonionic surfactant, especially where the level of quaternary ammonium compound is above about 8 % by weight of the total composition. Typically these can be included for the purpose of stabilising the compositions.

Suitable nonionic surfactants include addition products of ethylene oxide and/or propylene oxide with fatty alcohols, fatty acids and fatty amines. Any of the alkoxylated materials of the particular type described hereinafter can be used as the nonionic surfactant.

Suitable surfactants are substantially water soluble surfactants of the general formula:

$R\text{-}Y\text{-}(C_2H_4O)_z\text{-}CH_2\text{-}CH_2\text{-}OH$

where R is selected from the group consisting of primary, secondary and branched chain alkyl and/or acyl hydrocarbyl groups; primary, secondary and branched chain alkenyl hydrocarbyl groups; and primary, secondary and branched chain alkenyl-substituted phenolic hydrocarbyl groups; the hydrocarbyl groups having a chain length of from 8 to about 25, preferably 10 to 20, e.g. 14 to 18 carbon atoms.

In the general formula for the ethoxylated nonionic surfactant, Y is typically:

--O-- , --C(O)O-- , --C(O)N(R)-- or --C(O)N(R)R--

in which R has the meaning given above or can be hydrogen; and Z is at least about 8, preferably at least about 10 or 11.

Preferably the nonionic surfactant has an HLB of from about 7 to about 20, more preferably from 10 to 18, e.g. 12 to 16. Genapol™ C200 (Clariant) based on coco chain and 20 EO groups is an example of a suitable nonoionic surfactant.

The nonionic surfactant is present in an amount from 0.01 to 10%, more preferably 0.1 to 5 by weight, based on the total weight of the composition.

Antifoam

An antifoam may be present in an amount of from 0.025 to 0.45 wt %, preferably 0.03 to 0.4 wt %, most preferably from 0.05 to 0.35 wt %, for example 0.07 to 0.4 wt %, by weight of the total composition and based on 100 % antifoam activity.

A wide variety of materials may be used as antifoams, and antifoams are well known to those skilled in the art. See, for example, Kirk Othmer Encyclopedia of Chemical Technology, Third Edition, Volume 7, pages 430-447 (John Wiley & Sons, Inc., 1979).

Suitable antifoams include, for example, silicone antifoam compounds, alcohol antifoam compounds, for example 2-alkyl alcanol antifoam compounds, fatty acids, paraffin antifoam compounds, and mixtures thereof. By antifoam compound it is meant herein any compound or mixtures of compounds which act such as to depress the foaming or sudsing produced by a solution of a detergent composition, particularly in the presence of agitation of that solution.

Particularly preferred antifoam compounds for use herein are silicone antifoam compounds defined herein as any antifoam compound including a silicone component. Many such silicone antifoam compounds also contain a silica component. The term "silicone" as used herein, and in general throughout the industry, encompasses a variety of relatively high molecular weight polymers containing siloxane units and hydrocarbyl group of various types like the polyorganosiloxane oils, such as polydimethyl-siloxane, dispersions or emulsions of polyorganosiloxane oils or resins, and combinations of polyorganosiloxane with silica particles wherein the polyorganosiloxane is chemisorbed or fused onto the silica. Silica particles are often hydrophobed, e.g. as Trimethylsiloxysilicate. Silicone antifoam agents are well known in the art and are, for example, disclosed in U. S. Patent 4, 265, 779, issued May 5, 25 1981 to Gandolfo et al and European Patent Application No. 89307851. 9, published February 7, 1990, by Starch, M. S. Other silicone antifoams are disclosed in U. S. Patent 3, 455, 839 which relates to compositions and processes for defoaming aqueous solutions by incorporating therein small amounts of polydimethylsiloxane fluids. Mixtures of silicone and silanated silica are described, for instance, in German Patent Application DOS 2, 124, 526. Silicone defoamers and suds controlling agents in granular detergent compositions are disclosed in U. S. Patent 3, 933, 672, 35 Bartolotta et al, and in U. S. Patent 4, 652, 392, Baginski et al, issued March 24, 1987. Examples of suitable silicone antifoam compounds are the combinations of polyorganosiloxane with silica particles commercially available from Dow Corning, Wacker Chemie and Momentive.

Other suitable antifoam compounds include the monocarboxylic fatty acids and soluble salts thereof. These materials are described in US Patent 2, 954, 347. The monocarboxylic fatty acids, and salts thereof, for use as antifoam agents typically have hydrocarbyl chains of about 10 to about 24 carbon atoms, preferably about 12 to about 18 carbon atoms like the tallow amphopolycarboxyglycinate commercially available under the trade name TAPAC. Suitable salts include the alkali metal salts such as sodium, potassium, and lithium salts, and ammonium and alkanolammonium salts.

Other suitable antifoam compounds include, for example, high molecular weight hydrocarbons such as paraffin, light petroleum odourless hydrocarbons, fatty esters (e.g. fatty acid triglycerides, glyceryl derivatives, polysorbates), fatty acid esters of monovalent alcohols, aliphatic C18-C40 ketones (e.g. stearone) N-alkylated amino triazines such as tri- to hexa- 10 alkylmelamines or di- to tetra alkyldiamine chlortriazines formed as products of cyanuric chloride with two or three moles of a primary or secondary amine containing 1 to 24 carbon atoms, propylene oxide, bis stearic acid amide and monostearyl phosphates such as monostearyl alcohol phosphate ester and monostearyl di-alkali metal (e.g. , K, Na, and Li) phosphates and phosphate esters, and nonionic polyhydroxyl derivatives. The hydrocarbons, such as paraffin and 15 haloparaffin, can be utilized in liquid form. The liquid hydrocarbons will be liquid at room temperature and atmospheric pressure, and will have a pour point in the range of about -40°C and about 5°C, and a minimum boiling point not less than about 110°C (atmospheric pressure). It is also known to utilize waxy hydrocarbons, preferably having a melting point below about 100°C. Hydrocarbon suds suppressers are described, for example, in U. S. Patent 4, 265, 779. The hydrocarbons, thus, include aliphatic, alicyclic, aromatic, and heterocyclic saturated or unsaturated hydrocarbons having from about 12 to about 70 carbon atoms. The term "paraffin", as used in this suds suppresser discussion, is intended to include mixtures of true paraffins and cyclic hydrocarbons. Copolymers of ethylene oxide and propylene oxide, particularly the mixed ethoxylated/propoxylated fatty alcohols with an alkyl chain length of from about 10 to about 16 carbon atoms, a degree of ethoxylation of from about 3 to about 30 and a degree of propoxylation of from about 1 to about 10, are also suitable antifoam compounds for use herein.

Other antifoams useful herein comprise the secondary alcohols (e.g. , 2-alkyl alkanols as described in DE 40 21 265) and mixtures of such alcohols with silicone oils, such as the silicones disclosed in US 4,798,679, US 4,075,118 and EP 150,872. The secondary alcohols include the C6-C16 alkyl alcohols having a Cl-C16 chain like the 2-Hexyldecanol commercially available under the trade name ISOFOL16, 2-Octyldodecanol commercially available under the tradename ISOFOL20, and 2-butyl octanol, which is available under the trademark ISOFOL 12 from Condea. A preferred alcohol is 2-butyl octanol, which is available from Condea under the trademark ISOFOL 12. Mixtures of secondary alcohols are available under the trademark ISALCHEM 123 from Enichem. Mixed antifoams typically comprise mixtures of alcohol to silicone at a weight ratio of about 1:5 to about 5:1.

Further preferred antifoam agents are Silicone SRE grades and Silicone SE 47M, SE39, SE2, SE9 and SE10 available from Wacker Chemie; BF20+, DB310, DC1410, DC1430, 22210, HV495 and Q2-1607 ex Dow Corning; FD20P and BC2600 supplied by Basildon; and SAG 730 ex Momentive.

Other suitable antifoams, described in the literature such as in Hand Book of Food Additives, ISBN 0-566-07592-X, p. 804, are selected from dimethicone, poloxamer, polypropyleneglycol, tallow derivatives, and mixtures thereof. Preferred among the antifoams described above are the silicone antifoams, in particular the combinations of polyorganosiloxane with silica particles.

Non-ionic Ethoxylated Surfactant

A non-ionic ethoxylated surfactant may be present in order to improve the appearance of the rinse liquor. It prevents the formation of scum which could potentially lead to deposition of scummy deposits on the laundered fabric. In particular it disperses the reaction product of the anionic surfactant from the wash and monoquat compound preventing flocculation and formation of scum resulting in a translucent dispersion.

Suitable non-ionic surfactants are alkoxylated materials, particularly addition products of ethylene oxide and/or propylene oxide with fatty alcohols, fatty acids and fatty amines.

Preferred materials are of the general formula:

$$R\text{-}Y\text{-}(CH_2CH_2O)_zH$$

Where R is a hydrophobic moiety, typically being an alkyl or alkenyl group, said group being linear or branched, primary or secondary, and preferably having from 8 to 25, more preferably 10 to 20, and most preferably 10 to 18 carbon atoms; R may also be an aromatic group, such as a phenolic group, substituted by an alkyl or alkenyl group as described above; Y is a linking group, typically being O, CO.O, or CO.N(R^1), where R^1 is H or a $C_{1\text{-}4}$ alkyl group; and z represents the average number of ethoxylate (EO) units present, said number being 20 or more, preferably at least 25, more preferably at least 30.

Examples of suitable non-ionic surfactants include the ethoxylates of mixed natural or synthetic alcohols in the “coco” or “tallow” chain length. Preferred materials are condensation products of coconut fatty alcohol with 20-50 moles of ethylene oxide and condensation products of tallow fatty alcohol with 20-50 moles of ethylene oxide.

The ethoxylates of secondary alcohols such as 3-hexadecanol, 2-octadecanol, 4-eicosanol, and 5-eicosanol may also be used. Exemplary ethoxylated secondary alcohols have formulae C_{12}-EO(20); C_{14}-EO(20); C_{14}-EO(25); and C_{16}-EO(30). Suitable commercially available non-ionic surfactants include Lutensol AT25, Lutensol AT50 and Unitol CE 200F.

Optionally, the composition comprises an emulsifier that has an HLB of from 7 to 20, more preferably from 10 to 20, and most preferably from 15 to 20.

A particular surfactant may be useful in the present compositions alone or in combination with other surfactants. The preferred amounts of non-ionic surfactant indicated below refer to the total amount of such materials that are present in the composition.

The non-ionic surfactant is generally from 0.05 to 10%, usually 0.1 to 5%, and often 3 to 4% by weight, based on the total weight of the composition.

Further Optional Ingredients

The compositions of the invention may contain one or more other ingredients. Such ingredients include photobleaches, fluorescent agents, dyes, preservatives (e.g. bactericides), pH buffering agents, preferably inorganic or organic based such as hydrochloric acid, lactic acid and sodium lactate, etc, perfume carriers, hydrotropes, anti-redeposition agents, soil-release agents, polyelectrolytes, anti-shrinking agents, anti-wrinkle agents, anti-oxidants, sunscreens, anti-corrosion agents, drape imparting agents, anti-static agents and ironing aids.

It is believed that those polymers which deposit on cloth as a part of their activity may assist in the deposition of perfume components present. These include cationic polymeric deposition aids. Suitable cationic polymeric deposition aids include cationic guar polymers such as Jaguar™ (ex Rhone Poulenc), cationic cellulose derivatives such as Celquats™ (ex National Starch), Flocaid™ (ex National Starch), cationic potato starch such as SoftGel™ (ex Aralose), cationic polyacrylamides such as PCG (ex Allied Colloids).

Product Form

A composition for use in the invention is in liquid form. The composition may be a concentrate to be diluted, rehydrated and/or dissolved in a solvent, including water, before use. The composition may also be a ready-to-use (in-use) composition. Preferably the composition is provided as a ready to use liquid comprising an aqueous phase. The aqueous phase may comprise water-soluble species, such as mineral salts or short chain (C_{1-4}) alcohols.

The mineral salts may aid the attainment of the required phase volume for the composition, as may water soluble organic salts and cationic deflocculating polymers, as described in EP 41,698 A2 (Unilever). Such salts may be present at from 0.001 to 1% and preferably at from 0.005 to 0.1% by weight of the total composition. Examples of suitable mineral salts for this purpose include calcium chloride and magnesium chloride. The compositions of the invention may also contain pH modifiers such as hydrochloric acid. The short chain alcohols include primary alcohols, such as ethanol, propanol, and butanol, and secondary alcohols such as isopropanol. The short chain alcohol may be added with the cationic softening agent during the preparation of the composition.

The composition, being a fabric softener or fabric conditioner composition, is preferably for use in the rinse cycle of a home textile laundering operation, where, it may be added directly in an undiluted state to a washing machine, e.g. through a dispenser drawer or, for a top-loading washing machine, directly into the drum. Alternatively, it can be diluted prior to use. The compositions may also be used in a domestic hand-washing laundry operation.

Examples

Embodiments of the invention are now illustrated with reference to the following non-limiting examples. Unless stated otherwise, all proportions are given in weight percent by weight of the total composition.

Example 1: Preparation of Composition 1 and Comparative Examples A and B

Composition 1 and Comparative Example B both have the same composition but Composition 1 was prepared using the process of the invention, whilst Comparative Example B was prepared using the process of the prior art.

Table 1: Composition of Composition 1 and Comparative Examples A and B (wt %, by weight of total composition)

Ingredient		1	A	B
TEP-88L[1]	Fabric softening active	12-13.5	12-13.5	12-13.5
Benzalkonium Chloride	Water soluble quaternary ammonium surfactant	0.8	0.0	0.8
Lutensol AT25	Nonionic surfactant	0.1-2	0.1-2	0.1-2
Flosoft 270LS	Thickening polymer	0.1	0.1	0.1
1M HCL	pH adjustment	To required pH	To required pH	To required pH
Perfume encap slurry [2]	Encapsulated perfume	0.01	0.01	0.01
$CaCl_2$ (10% Soln.)	Electrolyte	0.0025	0.0025	0.0025
Perfume oil[2]		1.2 - 2.2	1.2 - 2.2	1.2 - 2.2
Water and minors[3]		To 100	To 100	To 100

[1] Palm based soft TEA Quat, ex FXG

[2] ex IFF

[3] antifoam, preservative, dye, pearlescer, sequestrant

Preparation of Composition 1

1. The fabric softening active and the non-ionic surfactant were heated to about 65°C to form a melt.
2. The water was heated to about 50°C with stirring.
3. The polymer was added to the water and mixed thoroughly.
4. Minor ingredients (preservative, sequestrant) were added.
5. The perfume encap slurry was then added with stirring.
6. Hydrochloric acid was added to the desired pH.
7. Benzalkonium Chloride was added to the water phase.

8. The melted premix was then added to the water phase.
9. Further minor ingredients and $CaCl_2$ were then added to the vessel.
10. The product was then cooled to 35°C.
11. Perfume oil and antifoam were then added to the cooled product with stirring.

Preparation of Comparative Example A (no Benzalkonium Chloride)
Comparative Example A was made in the same way as Composition 1 with the omission of Benzalkonium Chloride.

Preparation of Comparative Example B (Benzalkonium Chloride added at different stage)

Comparative Example B was made in the same way as Composition 1, only the Benzalkonium Chloride was added to the cooled product along with the perfume and antifoam and not before the addition of the melt.

Example 2: Storage Stability of Composition 1 and Comparative Examples A and B

The compositions were placed on storage at temperatures of 5, 20, 37 and 45 °C for up to 12 weeks. The viscosities of the formulations were measured after 4, 8 and 12 weeks storage.

Viscosity was measured on a cup and bob viscometer; the viscosity being continuously measured under shear at $106 s^{-1}$ for 60 seconds, at 25°C.

The results are shown in Tables 2-4 below.

Table 2: Viscosities (mPas-1) of Composition 1 upon storage at 5, 20, 37 and 45 °C for up to 12 weeks.

	initial	4wks	8wks	12wks
5°C	81.9	88.9	92.1	99
20°C	81.9	86.4	91.2	98
37°C	81.9	85.6	92.8	105
45°C	81.9	90.4	94.5	98

Table 3: Viscosity (mPas-1) of Comparative Example A upon storage at 5, 20, 37 and 45 °C for up to 12 weeks.

	initial	4wks	8wks	12wks
5°C	68	78	77	73
20°C	68	65	65	67
37°C	68	71	74	75
45°C	68	73	96	169

Table 4: Viscosity (mPas-1) of Comparative Example B upon storage at 5, 20, 37 and 45 °C for up to 12 weeks.

	initial	4wks	8wks	12wks
5°C	50	66	76	86
20°C	50	-	76	85
37°C	50	64	77	109
45°C	50	88	152	345

Much better high temperature stability was achieved in the composition prepared by the process in accordance with invention.

CLAIMS

1. A process for preparing an opaque, aqueous liquid fabric conditioning composition comprising

   (a) a water insoluble ester-linked quaternary ammonium softening active, which, in pure form as a strong acid salt, has a solubility in distilled water at pH 2.5 and 20°C of less than 1g/l, and
   (b) a water soluble quaternary ammonium surfactant, which is a cationic surfactant having a solubility in distilled water at pH 2.5 and 20°C of greater than 1g/l,
   (c) an encapsulated perfume,

   wherein the process comprises the step of dispersing the water soluble quaternary ammonium surfactant in water before the addition of the water insoluble ester-linked quaternary ammonium softening active to the water.

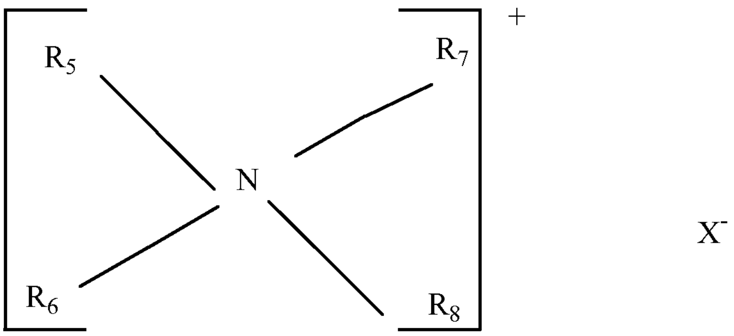
2. A process as claimed in claim 1, wherein the water insoluble ester-linked quaternary ammonium softening active conforms to the structure given by formula I:

   $[(CH_2)_n(TR)]_m\text{-}(R^1).N^+\text{-}[(CH_2)_n(OH)]_{3-m}\ X^-$ (I)

   wherein each R is independently selected from a C_{5-35} alkyl or alkenyl group;
   R^1 represents a C_{1-4} alkyl, C_{2-4} alkenyl or a C_{1-4} hydroxyalkyl group;
   T is generally O-CO., but may alternatively be CO.O;
   n is a number selected from 1 to 4;
   m is a number selected from 1, 2, or 3; and
   X^- is an anionic counter-ion.

3. A process as claimed in claim 2, wherein the water insoluble ester-linked quaternary ammonium softening active is a triethanolamine quaternary ammomium compound.

4. A process as claimed in any preceding claim, wherein the water soluble quaternary ammonium surfactant conforms to the structure given by formula II:



wherein:

R_5 represents a C_8-C_{24} alkyl or alkenyl group,

R_6 represents hydrogen, a C_1-C_{12} alkyl, alkenyl or hydroxyalkyl group, an aryl group, a C_{1-6} alkylaryl group, or a poly(ethylene oxide) group having from 2 to 20 ethylene oxide units,

R_7 and R_8 individually represent hydrogen, a C_1-C_4 alkyl, alkenyl or hydroxyalkyl group or a poly(ethylene oxide) group having from 2 to 20 ethylene oxide units; and

X is a counter-anion.

5. A process as claimed in claim 4, wherein the water soluble quaternary ammonium surfactant is benzalkonium chloride.

6. A process as claimed in any preceding claim, wherein the water insoluble ester-linked quaternary ammonium softening active is derived from fatty acyl or fatty acid feed stock having an Iodine Value of from 20 to 60.

7. A process as claimed in any preceding claim, wherein the water insoluble ester-linked quaternary ammonium softening active is present in the composition at a level of from 8 % to 16 %, by weight of the total composition.

8. A process as claimed in any preceding claim, which further comprises a non-encapsulated perfume.

9. A process as claimed in claim 7, wherein the total amount of non-encapsulated and encapsulated perfume is from 0.01 to 10 % by weight of the total composition.

10. A process as claimed in any preceding claim, which comprises the steps of:-

    1) mix the water soluble quaternary ammonium surfactant and perfume encaps, along with optional ingredients, with heated water to form a water phase;
    2) melt the water insoluble ester-linked quaternary ammonium softening active, and any optional non-ionic surfactant, to form a melt;
    3) combine the water phase and the melt with agitation;
    4) add dye, pearlescer and electrolyte if required;
    5) allow the resulting mixture to cool; and
    6) add any non-confined perfume oil and optional antifoam to the cooled mixture.

11. A composition obtained from the process of any one of the preceding claims.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2013/058501

A. CLASSIFICATION OF SUBJECT MATTER
INV. C11D1/645 C11D3/00 C11D11/00
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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A	WO 93/23510 A1 (PROCTER & GAMBLE) 25 November 1993 (1993-11-25) cited in the application examples IX,XI -----	1-11
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A	US 3 974 076 A (WIERSEMA YPE ET AL) 10 August 1976 (1976-08-10) example I -----	1-11

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

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"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

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Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer Hillebrecht, Dieter

INTERNATIONAL SEARCH REPORT

Information on patent family members

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