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54 **Fragrance carriers for laundry compositions.**

57 Fragrance compounds are dispersed in a matrix of solid non-ionic or cationic surfactant to obtain a fabric softener composition, usefully added to conventional laundry detergent compositions. The softergents so prepared, when used in normal laundering processes, result in an attachment of the fragrant softener component to the laundered textile fabric. The attachment survives a subsequent drying cycle so that the laundered and dried fabric remains associated with a slow-release fragrance.

Description

FRAGRANCE CARRIERS FOR LAUNDRY COMPOSITIONS

BACKGROUND OF THE INVENTION

Field of the Invention

The invention relates to laundry compositions and more particularly to a method and composition for providing a slow-release fragrance in a softergent composition.

Brief Description of the Prior Art

The prior art literature is replete with descriptions of compositions useful as fabric softeners and laundry detergents. Representative of such descriptions are those found in the U.S. Patents 4,145,184 (Brain et al, 1979); 4,259,373 (Demessemaekers et al., 1981); 4,292,035 (Battrell, 1981); 4,417,994 (Stoddart, 1983); 4,367,158 (Sprecker, 1983); 4,536,315 (Ramachandran et al, 1985); 4,536,316 (Ramachandran, 1985); and 4,539,135 (Ramachandran et al, 1985).

The extensive number of U.S. patents which have issued concerning the art is an indication of the dissatisfaction with earlier laundry compositions. There has been a constant demand for new and improved compositions and laundry methods. One problem which has resisted solution is retention of a desired fragrance adhered to laundered textiles, when the fragrance is added with the laundry softergent. It has been experimentally demonstrated that in a typical wash procedure only a trace amount of the originally incorporated fragrance in the softergent is retained on the cloth. This residual amount is further lost from the cloth during the drying operation. In order to develop a fragrance system that would minimize the losses, the following attributes are desirable:-

1. Transportation of fragrance to the textile fabric through the agency of a "Carrier" with a minimum loss of the fragrance despite the presence of detergents.
2. Once transported to the fabric, sufficient fragrance adherence to the fabric to survive wash and rinse cycles.
3. Affecting favorably the volatility of the fragrance to such an extent that the losses during the drying cycle are substantially minimized, providing the washed and dried fabrics with a strong retention of fragrance. Furthermore, the retained fragrance is then slowly released from the fabric. The invention disclosed herein provides the above described attributes and as such in an improvement in the art.

SUMMARY OF THE INVENTION

The invention comprises a solid composition of a non-ionic or a cationic surfactant having a volatile fragrance dispersed in the composition.

The term "volatile fragrance" means a compound which slowly volatilizes at room temperatures to provide a fragrant aroma.

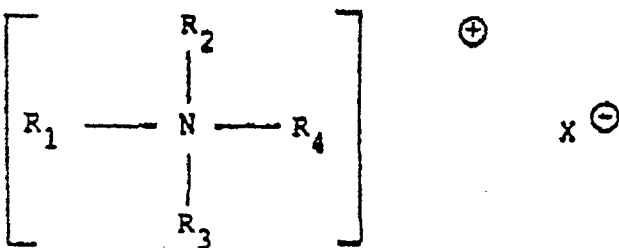
The invention also comprises a softergent composition which comprises the solid composition described above in admixture with a laundry detergent composition.

The term "softergent" as used herein means a laundering composition which comprises a laundry detergent and a fabric softener.

The compositions of the invention are useful in laundering textile fabrics.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS OF THE INVENTION

The cationic surfactants employed as a matrix for the fragrances in the compositions of the invention are solids (at room temperatures) selected from conventionally employed fabric softeners/antistat compounds, which are substantially water-insoluble (less than 1 percent solubility by weight at a temperature of 30°C.). Such compounds are inclusive of quaternary ammonium compounds and amines having at least one straight-chain organic groups of at least 8 carbon atoms. Preferred cation surfactant softener/antistat compounds are those within the structural formula:-



wherein R₁, R₂ and R₃ each independently represent hydrogen or hydrocarbyl of 1 to 22 carbon atoms,

inclusive; R₄ is alkyl or alkenyl of 8 to 22 carbon atoms, inclusive; and X is an anion selected from the group consisting of halogen, acetate, phosphate, nitrate, and methyl sulfate radicals.

The term "hydrocarbyl" as used herein means the monovalent moiety obtained upon removal of a hydrogen atom from a parent hydrocarbon. Representative of hydrocarbyl are alkyl of 1 to 22 carbon atoms, inclusive, such as methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, nonyl, undecyl, decyl, dodecyl, octadecyl, nonadecyl, eicosyl, heneicosyl, docosyl and the isomeric forms thereof; aryl of 6 to 22 carbon atoms, inclusive, such as phenyl, tolyl, xylyl, naphthyl, biphenyl and the like; aralkyl of 7 to 22 carbon atoms, inclusive, such as benzyl, phenethyl, phenpropyl, phenbutyl, phenhexyl, naphthoctyl and the like; cycloalkyl of 3 to 8 carbon atoms, inclusive, such as cyclopropyl, cyclobutyl, cyclopentyl, cyclohexyl, cycloheptyl, cyclooctyl and the like; alkenyl of 2 to 22 carbon atoms, inclusive, such as vinyl, allyl, butenyl, pentenyl, hexenyl, octenyl, nonenyl, decenyl, undecenyl, dodecyl, tridecenyl, pentadecenyl, octadecenyl and isomeric forms thereof.

Although not indicated in the above formula, R₁, R₂, R₃ and/or R₄ may be attached to the quaternary nitrogen atom through an ether, alkoxy, ester or amide linkage. Within the general description provided above concerning quaternary ammonium compounds useful for the invention are di-hydrogenated tallow dimethyl ammonium methyl sulfate; di-hydrogenated tallow dimethyl ammonium chloride, and 1-methyl-1-alkylamidoethyl-2-alkylimidazolium methyl sulfate wherein the "alkyls" are oleyl or saturated hydrocarbyls derived from tallow or hydrogenated tallow.

The quaternary ammonium salts employed herein are preferably substantially free of a conductive salt; the term "conductive salt" being used herein to refer to salts which are electrically conductive in aqueous solution. The conductive salts generally have a cation-anion bond of at least 50% ionic character as calculated in accordance with the method described in Pauling, "The Nature of the Chemical Bond", 3rd Edition, 1960. By use of the term "substantially free" is meant a concentration of conductive salt less than that present at normal impurity levels in the quaternary ammonium compound. Generally, the concentration of conductive salt is below 1%, by weight.

The non-ionic surfactants employed as a matrix for the fragrances in the compositions of the invention are also solids at room temperatures and are selected from known compounds which are substantially water-insoluble (less than 10 percent solubility by weight at a temperature of 30°C.). Representative of such non-ionic surfactants are the ethoxylated aliphatic alcohols. Such surfactants are well known as are methods of their preparation; see for example the extensive list of commercially available ethoxylated aliphatic alcohols given in the Kirk-Othmer Encyclopedia of Chemical Technology, Second Edition, Volume 19, Pages 538-539.

Volatile fragrances employed in the compositions of the invention include natural, essential oils and synthetic perfumes, and blends thereof.

The term "perfume" as used herein refers to odoriferous materials which are able to provide a pleasing fragrance to fabrics, and encompasses conventional materials commonly used in detergent compositions to counteract a malodor in such compositions and/or provide a pleasing fragrance thereto. The perfumes may be in the liquid state at ambient temperature, although solid perfumes are preferred. Included among the perfumes contemplated for use herein are materials such as aldehydes, ketones, esters and the like which are conventionally employed to impart a pleasing fragrance to liquid and granular detergent compositions. Naturally occurring plant and animal oils are also commonly used as components of perfumes. Accordingly, the perfumes useful for the present invention may have relatively simple compositions or may comprise complex mixtures of natural and synthetic chemical components, all of which are intended to provide a pleasant odor or fragrance when applied to fabrics. The perfumes used in detergent compositions are generally selected to meet normal requirements of odor, stability, price and commercial availability. A description of the materials conventionally used in detergent perfumery is set forth by R.T. Steltenkamp, in The Journal of The American Oil Chemists Society, Vol. 45, No. 6, pp.429-432, such disclosure being incorporated herein by reference.

Typical perfumery materials include: natural essential oils such as lemon oil, mandarin oil, clove leaf oil, petitgrain oil, cedar wood oil, patchouli oil, lavender oil, neroli oil, ylang oil, rose absolute or jasmin absolute; natural resins such as labdanum resin or olibanum resin; single perfumery chemicals which may be isolated from natural sources or manufactured synthetically, as for example alcohols such as geraniol, nerol, citronellol, linalool, tetrahydrogeraniol, beta-phenylethyl alcohol, methyl phenyl carbinol, dimethyl benzyl carbinol, menthol or cedrol; acetates and other esters derived from such alcohols; aldehydes such as citral, citronellal, hydroxycitronellal, lauric aldehyde, undecylenic aldehyde, cinnamaldehyde, amyl cinnamic aldehyde, vanillin or heliotropin; acetals derived from such aldehydes; ketones such as methyl hexyl ketone, the ionones and the methylionones; phenolic compounds such as eugenol and isoeugenol; and the like.

The fragrance is preferably substantially insoluble in water.

The compositions of the invention may be prepared by heating the solid surfactant to a temperature above its melting point and dispersing into the melt a fragrance emitting proportion of the volatile fragrance. A fragrance emitting proportion is generally within the range of from 1 to 60 percent by weight of the total cationic surfactant matrix. Conventional stirring apparatus may be used to obtain a homogenous dispersion. The dispersions may then be allowed to re-solidify by cooling to ambient, room temperatures. Advantageously the re-solidified composition is comminuated to obtain solid particles of a diameter which will pass through a No. 325 mesh screen (U.S. Sieve Series).

Alternatively, the solid surfactant can be pre-mixed with the volatile fragrance, and the mixture melted and re-solidified.

Also alternatively, the molten mixture of surfactant and volatile fragrance may be chill sprayed to obtain re-solidified particles of the desired size to avoid the otherwise necessary step of comminuating a larger solid.

The above-described compositions of the invention are advantageously employed as components of softergent laundry compositions by admixture with conventional laundry detergent compositions.

The conventional laundry detergent compositions with which the present fabric softening compositions of the invention may be incorporated may contain one or more surface active agents selected from the group consisting of anionic, nonionic, cationic, ampholytic and zwitterionic detergents. The synthetic organic detergents employed in the practice of the invention may be one or more of a wide variety of such compounds which are well known and are described at length in the text "Surface Active Agents and Detergents", Vol. II, by Schwartz, Perry and Berch, published in 1958 by Interscience Publishers, the relevant disclosure of which is hereby incorporated by reference.

The softergent compositions of the invention preferably employ one or more anionic detergent compounds as the primary detergent. The anionic detergent may be supplemented, if desired, with another type of detergent, preferably an ampholytic detergent. The use of a nonionic detergent is generally less preferred for the present invention, however, when used in combination with a detergent builder salt, nonionic detergents can be advantageously utilized.

Among the anionic detergents useful in the present invention are those surface active compounds which contain an organic hydrophobic group containing from about 8 to 26 carbon atoms and preferably from about 10 to 18 carbon atoms in their molecular structure and at least one water-solubilizing group selected from the group consisting of sulfonate, sulfate, carboxylate, phosphonate and phosphate so as to form a water-soluble detergent.

Examples of suitable anionic detergents include soaps, such as, the water-soluble salts (e.g., the sodium, potassium, ammonium and alkanol-ammonium salts) of higher fatty acids or resin salts containing from about 8 to 20 carbon atoms. Particularly useful are the sodium and potassium salts of the fatty acid mixtures derived from coconut oil and tallow, for example, sodium coconut soap and potassium tallow soap.

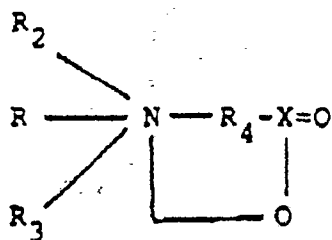
Other anionic detergents are the olefin sulfonates including long chain alkene sulfonates, long chain hydroxyalkane sulfonates or mixtures of alkene sulfonates and hydroxyalkane sulfonates. Also the sulfated ethoxylated higher fatty alcohols of the formula $RO(C_2H_4O)_mSO_3M$, wherein R is a fatty alkyl of from 10 to 18 carbon atoms, m is from 2 to 6 (preferably having a value from about 1/5 to 1/2 the number of carbon atoms in R) and M is a solubilizing salt-forming cation, such as an alkali metal, ammonium, lower alkylamino or lower alkanolamino, or a higher alkyl benzene sulfonate wherein the higher alkyls of 10 to 15 carbon atoms are present. The proportion of ethylene oxide in the polyethoxylated higher alkanol sulfate is preferably 2 to 5 moles of ethylene oxide groups per mole of anionic detergent, with three moles being most preferred, especially when the higher alkanol is of 11 to 15 carbon atoms.

The preferred water-soluble anionic detergent compounds are the ammonium and substituted ammonium (such as mono, di and tri-ethanolamine), alkali metal (such as, sodium and potassium) and alkaline earth metal (such as, calcium and magnesium) salts of the higher alkyl benzene sulfonates, olefin sulfonates and higher alkyl sulfates.

The nonionic organic detergents are characterized by the presence of an organic hydrophobic group and an organic hydrophilic group and are typically produced by the condensation of an organic aliphatic or alkyl aromatic hydrophobic compound with ethylene oxide (hydrophilic in nature). Practically any hydrophobic compound having a carboxy, hydroxy, amido or amino group with a free hydrogen attached to the nitrogen can be condensed with ethylene oxide or with the polyhydration product thereof, polyethylene glycol, to form a nonionic detergent. The length of the hydrophilic or polyoxyethylene chain can be readily adjusted to achieve the desired balance between the hydrophobic and hydrophilic groups.

The nonionic detergent is preferably a poly-lower alkoxyated higher alkanol wherein the alkanol is of 10 to 18 carbon atoms and wherein the number of moles of lower alkylene oxide (to 2 or 3 carbon atoms) is from 3 to 12. Of such materials it is preferred to employ those wherein the higher alkanol is a higher fatty alcohol of 11 to 15 carbon atoms and which contain from 5 to 9 lower alkoxy groups per mole. Preferably, the lower alkoxy is ethoxy but in some instances it may be desirably mixed with propoxy the latter, if present, usually being a minor (less than 50%) constituent. Exemplary of such compounds are those wherein the alkanol is of 12 to 15 carbon atoms and which contain about 7 ethylene oxide groups per mole, e.g., Neodol 25-7, and Neodol 23-6.5, which products are made by Shell Chemical Company, Inc. The former is a condensation product of a mixture of higher fatty alcohols averaging about 12 to 15 carbon atoms, with about 7 moles of ethylene oxide and the latter is a corresponding mixture wherein the carbon atom content of the higher fatty alcohol is 12 to 13 and the number of ethylene oxide groups per mole averages about 6.5. The higher alcohols are primary alkanols.

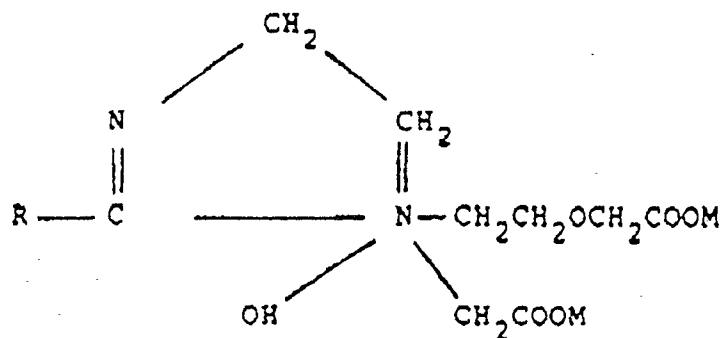
Zwitterionic detergents such as the betaines and sulfobetaines having the following formula are also useful:



wherein R is an alkyl group containing from about 8 to 18 carbon atoms, R₂ and R₃ are each an alkyl or hydroxyalkyl group containing about 1 to 4 carbon atoms, R₄ is an alkylene or hydroxyalkylene group containing 1 to 4 carbon atoms, and X is C or S:O. The alkyl group can contain one or more intermediate linkages such as amido, ether, or polyether linkages or nonfunctional substituents such as hydroxyl or halogen which do not substantially affect the hydrophobic character of the group. When X is C, the detergent is called a betaine; and when X is S:O, the detergent is called a sulfobetaine or sultaine.

Cationic surface active agents may also be employed as detergents. They comprise compounds which contain an organic hydrophobic group which forms part of a cation when the compound is dissolved in water, and an anionic group. Typical cationic detergents are amine and quaternary ammonium compounds. Examples of cationic detergents include: normal primary amines of the formula RNH₂ wherein R is an alkyl group containing from about 12 to 15 carbon atoms; diamines having the formula RNHC₂H₄NH₂ wherein R is an alkyl group containing from about 12 to 22 carbon atoms, such as N-2-aminoethyl-stearyl amine and N-2-aminoethyl myristyl amine; amide-linked amines such as those having the formula R₁CONHC₂H₄NH₂ wherein R₁ is an alkyl group containing about 8 to 20 carbon atoms, such as N-2-amino ethylstearyl amide and N-amino ethylmyristyl amide; quaternary ammonium compounds wherein typically one of the groups linked to the nitrogen atom is an alkyl group containing about 8 to 22 carbon atoms and three of the groups linked to the nitrogen atom are alkyl groups which contain 1 to 3 carbon atoms, including alkyl groups bearing inert substituents, such as phenyl groups, and there is present an anion such as halogen, acetate, methosulfate and the like. The alkyl group may contain intermediate linkages such as amide which do not substantially affect the hydrophobic character of the group, for example, stearyl amido propyl quaternary ammonium chloride. Typical quaternary ammonium detergents are ethyldimethylstearyl ammonium chloride, benzyldimethylstearyl ammonium chloride, trimethylstearyl ammonium chloride, trimethylcetyl ammonium bromide, dimethylethyl lauryl ammonium chloride, dimethylpropylmyristyl ammonium chloride, the corresponding methosulfates and acetates.

Amphotolytic detergents are well known in the art and many detergents of this class are disclosed by Schwartz, Perry and Berch in the aforementioned "Surface Active Agents and Detergents." Examples of amphoteric detergents include: alkyl betainodipropionates and long chain imidazole derivatives having the general formula:



wherein R is an acyclic hydrophobic group containing from about 8 to 18 carbon atoms and M is a cation to neutralize the charge of the anion. Specific amphoteric detergents include the disodium salt of undecyclomidiniummethoxyethionic acid-2-ethionic acid, dodecyl beta alanine, and the inner salt of 2-trimethylamino lauric acid.

The softergent compositions of the invention optionally contain a detergent builder of the type commonly used in detergent formulations. Useful builders include any of the conventional inorganic water-soluble builder salts, such as, for example, water-soluble salts of phosphates, pyrophosphates, orthophosphates, polyphosphates, silicates, carbonates, and the like. Organic builders include water-soluble phosphonates, polyphosphonates, polyhydroxysulfonates, polyacetates, carboxylates, polycarboxylates, succinates and the like.

Specific examples of inorganic phosphate builders include sodium and potassium tripolyphosphates, pyrophosphates and hexametaphosphates. The organic polyphosphonates specifically include, for example, the sodium and potassium salts of ethane, 1-hydroxy-1, 1-diphosphonic acid and sodium and potassium salts of ethane-1,1,2-triphosphonic acid. Examples of these are other phosphorous builder compounds are disclosed in the U.S. Pat. Nos. 3,213,030; 3,422,021; 3,422,137 and 3,400,176.

	<u>Parts</u>	
sodium dodecyl	15.0	
benzene sulfonate		
Neodol 25-7*	5.0	5
sodium	30.0	
tripolyphosphate		
sodium silicate	10.0	
sodium sulfate	39.8	
carboxymethyl	<u>0.2</u>	10
cellulose		
	100.0	

* The condensation product of a mixture of C₁₂-C₁₅ Average alcohols ethoxylated with 7 moles of ethylene oxide/mole of alcohol, Shell Chemical Co., Inc. 15

The following Examples 1-3 are not examples of the invention but are made for comparative purposes. 20

Example 1

The detergent composition of Preparation, 1 supra., (99.5 gms) was blended with 0.5 gms of the fragrance 3350D* in a blender for 30 minutes. This preparation is a typical marketed detergent product. * Bush, Boake, Allen, Inc. 25

Example 2

A blend of the detergent composition of preparation 1, supra. (94.5 gms) and benzalkonium chloride (5.0 gms) was mixed further with 0.5 gms of fragrance 3350D supra., in a blender for 30 minutes. This product represents a typical softergent. 30

Example 3

This formulation was put together according to the method of the Example 1 described in U.S. Patent No. 4,536,315 but using the fragrance 3350D, supra. The composition is a softergent on particles of a smectite-type of clay with an adhesive agent. 35

Example 4

An encapsulated fragrance 3350D, Supra. was prepared as follows:- Solid benzalkonium chloride (90.0 gms) was mixed with the fragrance and the mixture was then heated to 110°C until completely molten. The molten solution of the fragrance is stirred and chill sprayed through a nozzle using a Bowman spray dryer. The product collected as a free flowing solid which had a fragrance content of 10% by weight. 40

The detergent formulation of preparation 1, supra. (5.0 gms.) was then blended with the encapsulated fragrance (5.0 gms). 45

The compositions prepared according to the Examples 1-4 were then tested for fragrance strength and character as described above. 45

The scores from the individual panelists were averaged and results tabulated as follows:-

	<u>EXAMPLE</u>				
	1	2	3	4	
STRENGTH	2.8	3.0		4	6.5
CHARACTER	2.0	2.5		5	8.0

From the test results it may be observed that:

1. In comparison to the powder detergent products generally available in the marketplace, the detergent product of the compositions of the invention minimize the loss of fragrance during the wash and rinse cycle. The compositions of the invention suffer minimum loss of fragrance during washing, despite the heavy concentration of detergents present in the wash water. Furthermore, it also provides protection from loss through evaporation during the drying cycle. 55

2. Fabrics washed with the composition of the invention do not require the additional use of either a liquid fabric softener in the washer or a fabric softener sheet in the dryer. The quaternary compound used to encapsulate the fragrance is strongly retained by the fabric. In the process the fragrance is also adhered strongly to the fabric and emanates from the fabric with a pleasing and longer lasting effect. Therefore, the combining of the detergent and the fabric softener in one product with the retention of desirable levels of fragrance throughout the wash/dry is obtained. 60 65

3. The composition of Example 3 is largely based on absorbing the fragrance into a clay matrix. But clay, with its trace metal impurities, is hostile towards a large number of fragrance materials rendering them undesirable and, hence, imposing restriction in the creation of a wide range of fragrance types. Also, once incorporated into the clay, the fragrance is held strongly in the matrix, thus suppressing the release of the fragrance.

Example 5

A mixture of 85 parts by weight of a non-ionic surfactant (Unithox 720, a C₃₀-C₅₀ alkylpoly(ethyleneoxy) ethanol; Petrolite Co.) and 15 parts by weight of the fragrance 3350 D, supra. was heated to a temperature of 90°C. and the molten mixture then spray chilled through a nozzle using a Bowman spray dryer. The product was collected as a free-flowing powder with a 15 percent fragrance content. A detergent powder formulation was then prepared according to the following recipe:

	<u>Ingredient</u>	<u>Parts by Weight</u>
15	Sodium Dodecyl Benzene Sulfonate	15.0
	Neodol 25-7, supra.	5.0
	Sodium Tripolyphosphate	30.0
20	Sodium Silicate	10.0
	Sodium Sulfate	36.5
	Carboxymethyl Cellulose	0.2
25	Encapsulated Fragrance	3.3
		100.0

30 The powder formulation, is tested as described above, for fragrance strength and character. The scores from the individual panelists were averaged and the results tabulated as follows, in comparison to the average determined for the unencapsulated fragrance (neat oil).

STRENGTH

Scale:

0----1----2----3----4----5----6----7----8----9----10

Absent

Moderate

Very
Strong

Encapsulated composition - 6.0

Neat Oil, unencapsulated - 3.5

CHARACTER

Scale

0----1----2----3----4----5----6----7----8----9----10

Absent

Good

Excellent

Encapsulated composition - 7.0

Neat Oil, unencapsulated - 3.0

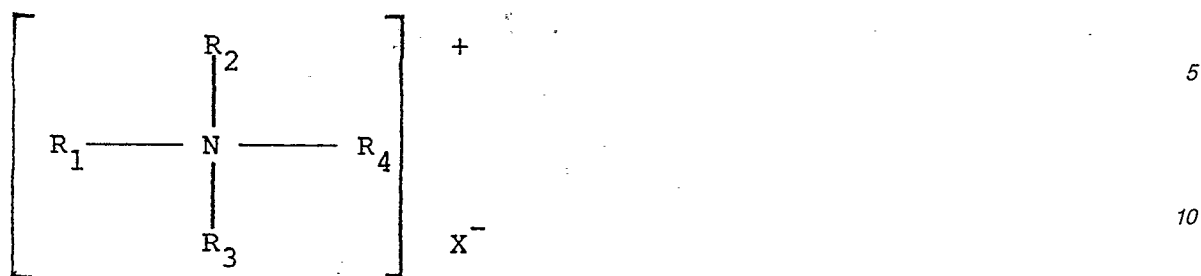
Claims

1. A solid composition which comprises a solid non-ionic or cationic surfactant having a volatile fragrance dispersed in the solid.

2. The composition of claim 1 wherein the surfactant is a cationic surfactant, preferably having a

solubility in water of less than 1% at 30°C.

3. The composition of claim 2 wherein the surfactant is selected from those of the formula:



wherein R₁, R₂ and R₃ each independently represent hydrogen or hydrocarbyl of 1 to 22 carbon atoms, inclusive; R₄ is alkyl or alkenyl of 8 to 22 carbon atoms, inclusive; and X⁻ is an anion selected from the group consisting of halogen, acetate, phosphate, nitrate and methyl sulfate, and is preferably benzalkonium chloride.

4. The composition of claim 1 wherein the surfactant is a non-ionic surfactant, preferably having a solubility in water of less than 10% at a temperature of 30°C.

5. The composition of claim 4 wherein the surfactant is an alkylpoly(ethyleneoxy) alcohol, preferably a C₃₀-C₅₀ alkylpoly(ethyleneoxy) ethanol.

6. The composition of any preceding claim wherein the fragrance comprises from 1 to 60 percent by weight of the total composition.

7. The composition of any preceding claim wherein the fragrance is substantially water-insoluble.

8. A softergent composition comprising the composition of any preceding claim in admixture with a laundry detergent composition.

9. A process for producing a solid composition which comprises a solid non-ionic or cationic surfactant having a volatile fragrance dispersed in the solid, in which the fragrance in particulate form and surfactant are mixed at a temperature at which the surfactant is liquid and the mixture is cooled to a temperature below the melting point of the surfactant.

10. A process according to claim 9 in which the dispersion of fragrance in molten surfactant is cooled by being chill sprayed to form particles of the composition.