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(54) **VISCOSITY STABLE CONCENTRATED LIQUID FABRIC SOFTENER COMPOSITIONS**

VISKOSITÄTSSTABILE KONZENTRIERTE WEICHSPÜLERZUSAMMENSETZUNGEN

COMPOSITIONS ASSOUPPLISSANTES CONCENTREES LIQUIDES, DE VISCOSITE STABLE

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DescriptionTECHNICAL FIELD

[0001] The present invention relates to concentrated aqueous textile treatment compositions. In particular, it relates to textile treatment compositions for use in the rinse cycle of a textile laundering operation to provide fabric softening/static control benefits, the compositions being characterized by excellent storage stability and excellent viscosity stability after freeze/thaw cycling.

BACKGROUND OF THE INVENTION

[0002] Aqueous textile treatment compositions suitable for providing fabric softening and static control benefits during laundering are well-known in the art and have found wide-scale commercial application. Conventionally, aqueous, rinse-added, fabric softening compositions contain, as the active softening component, substantially water-insoluble cationic materials having two long alkyl chains. Typical of such materials are di-hydrogenated tallow di-methyl ammonium chloride and imidazolinium compounds substituted with two stearyl groups. These materials are normally prepared in the form of a dispersion in water. It is generally not possible to prepare such aqueous dispersions with more than about 10% cationic materials without encountering intractable problems of product viscosity and stability, especially after storage at lower temperatures, such that the compositions are unpourable and have inadequate dispensing and dissolving characteristics in rinse water. This physical restriction on softener concentration limits the level of softening performance achievable without using excessive amounts of product and also adds substantially to the costs of distribution and packaging. Accordingly, it would be highly desirable to prepare physically acceptable aqueous textile treatment compositions containing much higher levels of substantially water-insoluble cationic softener materials.

[0003] Cationic softener materials are normally supplied by the manufacturer containing about 70%-90% of active material in an organic liquid such as isopropanol or ethanol, sometimes containing a minor amount of water (up to 10%). Retail fabric softening compositions are then prepared by dispersion of the softener in warm or hot water under carefully controlled conditions. The physical form and dispersibility constraints of these industrial concentrates are such as to preclude their direct use by the domestic consumer; indeed, they can pose severe processing problems even for the industrial supplier of retail fabric softening compositions.

[0004] Many of the various solutions to the specific problem of preparing aqueous fabric softening compositions in concentrated form suitable for consumer use have not been entirely satisfactory. It is generally known (for example, in U.S. Pat. No. 3,681,241, Rudy, issued Aug. 1, 1972) that the presence of ionizable salts in softener compositions does help reduce viscosity, but this approach by itself is ineffective in preparing compositions containing more than about 12% of dispersed softener, inasmuch as the level of ionizable salts necessary to reduce viscosity to any substantial degree has a seriously detrimental effect on product viscosity stability.

WO 93/23510 describes storage stable fabric softener composition containing biodegradable fabric softeners and a single long chain cationic surfactant.

EP-A-0,643,128, which is a document according to Art. 54(3) EPC, discloses aqueous fabric softener compositions comprising a quaternary ammonium salt with an amido ester link.

WO 94/20597, which is a document according to Art. 54(3) EPC, discloses a fabric softening composition comprising biodegradable quaternary ammonium fabric softener compositions with an intermediate iodine Value.

SUMMARY OF THE INVENTION

[0005] It has now been discovered that the product stability and viscosity characteristics of concentrated fabric softener compositions containing mixtures of (A) biodegradable diester quaternary ammonium softening materials, (hereinafter referred to as "(A)"), and (B) specific co-active fabric softening materials, (hereinafter referred to as "(B)"), (i.e., specific quaternary ammonium salts which are not the same as (A)), are superior both at normal and lower temperatures. The value of using such mixtures of fabric softening materials for enhancing the long term viscosity characteristics and stability of concentrated aqueous cationic fabric compositions especially after freeze/thaw cycling, has hitherto not been recognized in the art

DETAILED DESCRIPTION OF THE INVENTION(A). Biodegradable Quaternized Ester-Amine Softening Material

[0006] The present invention contains diester quaternary ammonium material (hereinafter referred to as "DEQA")

as an essential component. Two primary types of DEQA are preferred.

[0007] 1. The first type of DEQA preferably comprises, as the primary active, compounds of the formula:



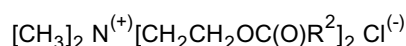
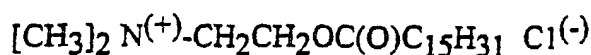
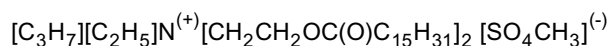
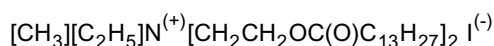
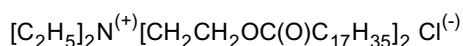
wherein each R substituent is a short chain C_1-C_6 , preferably C_1-C_3 alkyl group, e.g., methyl (most preferred), ethyl, propyl, and the like, benzyl or mixtures thereof each m is 2; each n is from 1 to about 4; each Y is $-O-(O)C-$, or $-C(O)-O-$, but not $-OC(O)O-$; each R^2 is a long chain $C_{12}-C_{22}$ hydrocarbyl, or substituted hydrocarbyl substituent, preferably $C_{15}-C_{19}$ alkyl or alkylene, most preferably $C_{15}-C_{17}$ straight chain alkyl or alkylene such that the Iodine Value (hereinafter referred to as IV) of the parent fatty acid of this R^2 group is less than about 10, preferably less than about 5, most preferably less than about 2; and the counterion, $X^{(-)}$, can be any softener-compatible anion, preferably the anion of a strong acid, for example, chloride, bromide, methylsulfate, formate, sulfate, nitrate and the like. The anion can also, but less preferably, carry a double charge in which case $X^{(-)}$ represents half a group. These materials containing a divalent anion, in general, are more difficult to formulate as stable concentrated liquid compositions.

[0008] Carbonate esters, i.e., where $Y = -O-C(O)O-$, are unstable compounds and are not included as (A)(1) compounds.

[0009] It will be understood that substituent R^2 can optionally be substituted with various groups such as alkoxy or hydroxyl groups, and can be straight, or branched so long as the R^2 groups maintain their basically hydrophobic character. The preferred compounds can be considered to be diester variations of ditallow dimethyl ammonium chloride (hereinafter referred to as "DTDMAC"), which is a widely used fabric softener. At least 80% of the DEQA is in the diester form, and from 0% to 20% can be DEQA monoester (e.g., only one of the ester groups is hydrolyzed to yield either $-H$, or an $-OH$ group).

[0010] As used herein, when the diester is specified, it can include the monoester that is present. For softening, under no/low detergent carry-over laundry conditions the percentage of monoester should be as low as possible, preferably no more than about 2.5%. However, under high, anionic detergent surfactant or detergent builder carryover conditions, some monoester can be preferred. The overall ratios of diester to monoester are from 100:1 to 2:1, preferably from 50:1 to 5:1, more preferably from 13:1 to 8:1. Under high detergent carry-over conditions, the di/monoester ratio is preferably about 11:1. The level of monoester present can be controlled in manufacturing the DEQA.

[0011] The above compounds, used as the biodegradable quaternized ester-amine softening material in the practice of this invention, can be prepared using standard reaction chemistry. In one synthesis of a di-ester variation of DTDMAC, an amine of the formula $RN(CH_2CH_2OH)_2$ is esterified at both hydroxyl groups with an acid chloride of the formula $R^2C(O)Cl$, then quaternized with an alkyl halide, RX , to yield the desired reaction product (wherein R and R^2 are as defined hereinbefore). However, it will be appreciated by those skilled in the chemical arts that this reaction sequence allows a broad selection of agents to be prepared. The following are non-limiting examples (wherein all long-chain alkyl substituents are straight-chain):

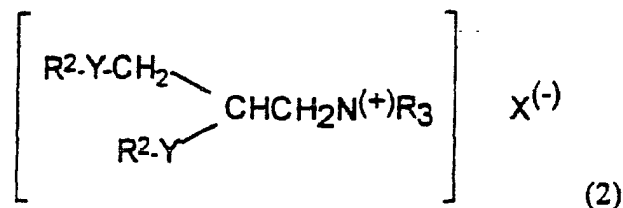


where $-C(O)R^2$ is derived from hardened tallow fatty acid.

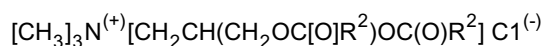
[0012] Since the foregoing materials (diesters) are somewhat labile to hydrolysis, they should be handled rather carefully when used to formulate the fabric softening composition herein. For example, stable liquid compositions herein are formulated at a neat pH in the range of from 2.5 to 4. The pH can be adjusted by the addition of a Bronsted acid. pH ranges for making stable softener compositions containing diester quaternary ammonium fabric softening compounds are disclosed in U.S. Pat. No. 4,767,547, Straathof, issued Aug. 30, 1988.

[0013] Examples of suitable Bronsted acids include the inorganic mineral acids, carboxylic acids, in particular the low molecular weight (C_1 - C_5) carboxylic acids, and alkylsulfonic acids. Suitable inorganic acids include HCl, H_2SO_4 , HNO_3 and H_3PO_4 . Suitable organic acids include formic, acetic, citric, methylsulfonic and ethylsulfonic acids. Preferred acids are hydrochloric, phosphoric, and citric acids.

[0014] 2. In a second type of DEQA the primary active has the general formula:



wherein each Y, R, R^2 , and $X^{(-)}$ have the same meanings as before. Such compounds include those having the formula:



where $C(O)R^2$ is derived from hardened tallow fatty acid.

[0015] Preferably each R is a methyl or ethyl group and preferably each R^2 is in the range of C_{15} to C_{19} . Degrees of branching and substitution can be present in the alkyl chains. The anion $X^{(-)}$ in the molecule is the same as in DEQA (1) above. As used herein, when the diester is specified, it can include the monoester that is present. The amount of monoester that may be present is the same as in DEQA (1).

[0016] The present invention may also contain mixtures of the two primary types of DEQA.

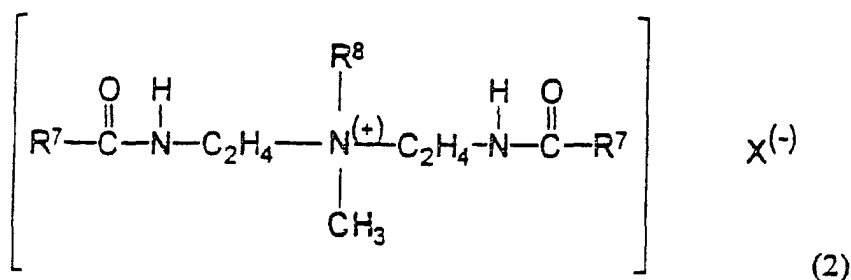
[0017] These types of agents and general methods of making them are disclosed in U.S. Pat. No. 4,137,180, Naik et al, issued Jan. 30, 1979.

(B). The Co-Active Fabric Softening Material

[0018] Compositions prepared by the present invention contain as an essential component a co-active fabric softening material, as described hereinafter, which is different from the biodegradable diester quaternary ammonium softening material

(A):

[0019] 2. The Di(2-amidoethyl)methyl quaternary ammonium salts are also suitable for use as Component (B) in the compositions of the invention herein, especially those having the formula:

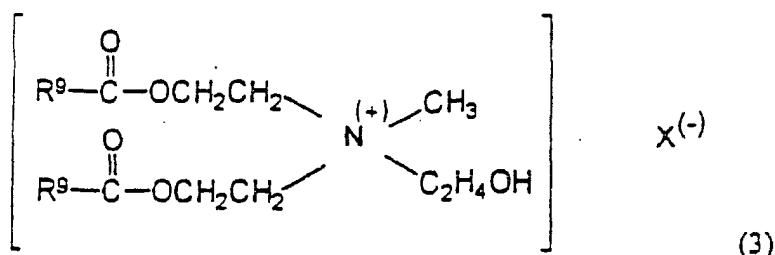


wherein each R^7 is selected from the group consisting of C_{14} to C_{20} alkyl and alkenyl groups, wherein each R^8 is selected from methyl, ethyl, and $-(C_mH_{2m}O)_n3H$, wherein n^3 is from 1 to about 5, preferably 3, and wherein m , and $X^{(-)}$ have the same meaning as before. This class of agents is disclosed in U.S. Pat. No. 4,134,840. Minegishi et al., issued January 16, 1979.

[0020] Exemplary materials are di((2-hydrogenatedtallowamidoethyl) ethoxylated (2 ethoxy groups) methyl ammonium methylsulfate, di(2-oleylamidoethyl) propoxylated (3-propoxy groups) methyl ammonium bromide, di(2-palmitoleylamidoethyl) dimethyl ammonium ethylsulfate and di(2-stearylamoethyl) propoxylated (2 propoxy groups) methyl ammonium methylsulfate.

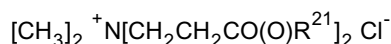
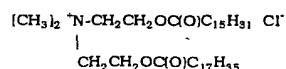
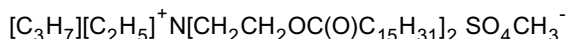
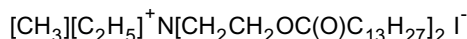
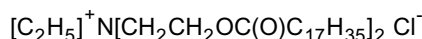
[0021] An exemplary commercial material suitable for use as Component (B) herein is di(2-tallowamidoethyl) ethoxylated methyl ammonium methylsulfate (such that the I.V. Component B is about 31) sold under the name Varisoft 222, from Witco Chemical Company.

[0022] 3. A co-active fabric softening material for use as component (B) in the composition of the invention herein can also have the formula:



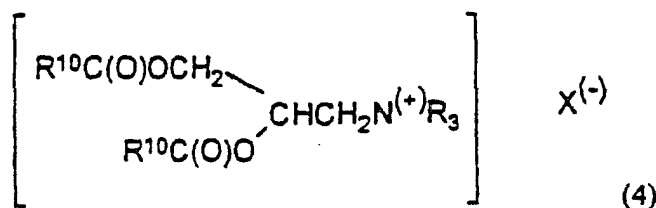
wherein each R^9 is C_{15} - C_{17} alkyl group such that the IV of the parent fatty acid of this R^9 group is from 20 to 100, preferably from 30 to 70, most preferably from 35 to 60; and $X^{(-)}$ has the same meaning as before.

[0023] A preferred fabric softening material of the present invention is prepared according to the synthesis disclosed in U.S. Pat. No. 3,915,867, Kang et al., issued October 28, 1975. The fabric softening material generally comprises the reaction of purified C_{14} - C_{18} fatty acid alkylester mixture, triethanolamine, and a quaternizing reagent, preferably dimethyl sulfate. The select fatty acid alkylesters are preferably a mixture of substantial amounts of oleic, palmitic, stearic add alkyl esters and may include minor amounts of other fatty substances. When B(3) is $[CH_2CH_2OH] [CH_3]^+N [CH_2CH_2OC(O)R^2]_2 X^-$, X^- being as hereinbefore defined, A is not (1), and wherein when B (3) is $[CH_2CH_2OH] [CH_3]^+N[(CH_2CH_2OC(O)R^{20}) Cl^-]$, where $-C(O)R^{20}$ is derived from partially hydrogenated tallow or modified tallow. X^- being as hereinbefore defined. A is not



where $-CO(O)R^{21}$ is derived from saturated tallow;

[0024] 4. A co-active fabric softening material suitable for use as Component (B) in the composition of the invention can also have the formula:



wherein each R^{10} is a C_{12} - C_{22} hydrocarbyl or substituted hydrocarbyl substituent, preferably C_{15} - C_{19} alkyl or alkylene, most preferably C_{15} - C_{17} straight chain alkyl or alkylene such that the IV of the parent fatty acid of this R^{10} group is from 20 to 100, preferably from 30 to 70, most preferably from 35 to 60; and R and $\text{X}^{(-)}$ have the same meaning as before.

[0025] Tallow is a convenient and inexpensive source of long chain alkyl and alkenyl materials.

[0026] A specific example of a diester quaternary ammonium compound suitable for use in this invention herein include:

1,2-ditallowyloxy-3-(trimethyl ammonio)propane chloride.

[0027] Other examples of suitable diester quaternary ammoniums of this invention are obtained by, e.g.: replacing "tallowyl" in the above compounds with, for example, cocoyl, palmoyl, lauryl, oleyl, stearyl, palmityl, or the like; replacing "methyl" in the above compounds with ethyl, propyl, isopropyl, butyl, isobutyl, t-butyl, or the hydroxy substituted analogs of these radicals; replacing "chloride" in the above compounds with bromide, methylsulfate, formate, sulfate, or nitrate.

[0028] In fact, the anion is merely present as a counterion of the positively charged quaternary ammonium compounds disclosed herein. The scope of this invention is not considered limited to any particular anion. When the DEQA (A) material is (2), the requirement for component (B) is not met by this material and it is not present, unless a third softener active is present in the mixture.

[0029] The materials herein can be prepared by standard esterification and quaternization reactions, using readily available starting materials. General methods for preparation are disclosed in U.S. Pat. No. 4,137,180.

[0030] 5. A co-active fabric softening material suitable for use as Component (B) in the composition of the invention can also have the formula:



except for those according to (3), wherein each R^{11} is a long chain C_{12} - C_{22} , hydrocarbyl, or substituted hydrocarbyl substituent, preferably C_{15} - C_{19} alkyl or alkylene, most preferably C_{15} - C_{17} straight chain alkyl or alkylene such that the IV of the parent fatty acid of this R^{11} group is from 20 to 100, preferably from 30 to 70, most preferably from 35 to 60; and each m, n, Y, R and $\text{X}^{(-)}$ have the same meaning as before. When the DEQA (A) material is (1), the requirement for component (B) is not met by this material and it is not present

[0031] The present invention may also contain mixtures of the various co-active fabric softening materials.

[0032] The compositions of the present invention herein comprise from about 15% to about 35%, preferably from about 20% to about 32%, most preferably from about 22% to about 27% of component (A) + component (B).

[0033] The ratio of component (A) to component (B) is from 0.25:1 to 4:1, preferably from 0.3:1 to 1.5:1.

(C). The Acid Component

[0034] The present invention utilizes an acid of sufficient concentration to keep the pH at the desired level and optionally, to fully protonate component (B) to the extent that it is not already quaternized. The composition of the present invention is prepared using a molten premix of components (A) + (B), (hereinafter referred to as premix). The premix is injected into an acid/water seat, then high shear milling is conducted and, electrolyte is added in no specific order. The electrolyte is selected from the group consisting of the Group IA and IIA metals of the periodic table of elements, e.g., calcium chloride, sodium chloride, potassium bromide, and lithium chloride, and ammonium salts, e.g., ammonium chloride and lysine HCl. Typically, the acid/water seat has an acid concentration up to about 2%.

[0035] Typically the acid to (B) molar ratio is from about 0:1 to about 1.2:1. Typically the acid to (A) molar ratio is from about 0:1 to about 0.2:1. The neat pH of the final composition is preferably from about 2.5 to about 4.

[0036] Suitable acids include the Bronsted acids, especially inorganic mineral acids and organic acids such as car-

boxylic acids. Carboxylic acids include, in particular, the low molecular weight (C_1 - C_5) carboxylic acids of the formula R^{12} -COOH (R^{12} being a C_1 - C_5 or H alkyl group). Suitable organic acids are selected from the group having the formula $R^{13}CH_2SO_3H$, wherein R^{13} is hydrogen or C_1 to C_4 alkyl. Suitable specific organic acids include formic, methylsulfonic, ethylsulfonic, citric, gluconic, and aromatic carboxylic acids like benzoic acid. Suitable inorganic acids include HCl, HBr, H_2SO_4 , H_2SO_3 , HNO_3 , and H_3PO_4 .

[0037] Preferred acids are phosphoric, formic, acetic, hydrochloric, citric, and methylsulfonic acids. Mixtures of the above organic and inorganic acids are also suitable. Typically, acids such as citric, hydrochloric, phosphoric, and sulfuric are used because of their low cost and availability.

(D). Liquid Carrier

[0038] The compositions of the present invention herein comprise from about 60% to about 90%, preferably from about 65% to about 85% of an aqueous liquid carrier. The preferred aqueous carrier is water which can contain minor ingredients.

(E). Optional Ingredients

[0039] Fully-formulated fabric softening compositions made by the process of the present invention can optionally contain one or more of the following ingredients.

1. Silicone Component

[0040] The fabric softening compositions herein optionally contain an aqueous emulsion of a predominantly linear polydialkyl or alkyl aryl siloxane in which the alkyl groups can have from one to five carbon atoms and can be wholly, or partially, fluoridated. These siloxanes act to provide improved fabric benefits. Suitable silicones are polydimethyl siloxanes having a viscosity, at 25°C, of from about 100 to about 100,000 centistokes, preferably from about 1,000 to about 12,000 centistokes. In some applications as low as 1 centistoke materials are preferred.

[0041] The fabric softening compositions herein can contain from about 0.1% to about 10%, of the silicone component

2. Thickening Agent

[0042] Optionally, the fabric softening compositions herein contain from 0% to about 3%, preferably from about 0.01% to about 2%, of a thickening agent. Examples of suitable thickening agents include: cellulose derivatives, synthetic high molecular weight polymers (e.g., carboxyvinyl polymer and polyvinyl alcohol), and cationic guar gums.

[0043] The cellulosic derivatives that are functional as thickening agents herein can be characterized as certain hydroxyethers of cellulose, such as Methocel, marketed by Dow Chemicals, Inc.; also, certain cationic cellulose ether derivatives, such as Polymer JR-125, JR-400, and JR-30M, marketed by Union Carbide.

[0044] Other effective thickening agents are cationic guar gums, such as Jaguar Plus, marketed by Stein Hail, and Gendrive 458, marketed by General Mills.

[0045] Preferred thickening agents herein are selected from the group consisting of methyl cellulose, hydroxypropyl methylcellulose, hydroxybutyl methylcellulose, or mixtures thereof, said cellulosic polymer having a viscosity in 2% aqueous solution at 20°C of from about 15 to about 75,000 centipoises.

3. Soil Release Agent

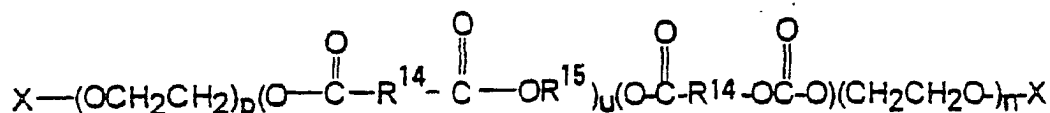
[0046] In the present invention, an optional soil release agent may be added. The addition of the soil release agent may occur in combination with the premix, in combination with the acid/water seat, before or after electrolyte addition, or after the final composition is made. The softening composition prepared by the process of the present invention herein can contain from 0% to about 10%, preferably from 0.2% to about 5%, of a soil release agent. Preferably, such a soil release agent is a polymer. Polymeric soil release agents useful in the present invention include copolymeric blocks of terephthalate and polyethylene oxide or polypropylene oxide, and the like.

[0047] A preferred soil release agent is a copolymer having blocks of terephthalate and polyethylene oxide. More specifically, these polymers are comprised of repeating units of ethylene terephthalate and polyethylene oxide terephthalate at a molar ratio of ethylene terephthalate units to polyethylene oxide terephthalate units of from 25:75 to about 35:65, said polyethylene oxide terephthalate containing polyethylene oxide blocks having molecular weights of from about 300 to about 2000. The molecular weight of this polymeric soil release agent is in the range of from about 5,000 to about 55,000.

[0048] Another preferred polymeric soil release agent is a crystallizable polyester with repeat units of ethylene tereph-

thalate units containing from about 10% to about 15% by weight of ethylene terephthalate units together with from about 10% to about 50% by weight of polyoxyethylene terephthalate units, derived from a polyoxyethylene glycol of average molecular weight of from about 300 to about 6,000, and the molar ratio of ethylene terephthalate units to polyoxyethylene terephthalate units in the crystallizable polymeric compound is between 2:1 and 6:1. Examples of this polymer include the commercially available materials Zelcon 4780 (from Dupont) and Milease T (from ICI).

[0049] Highly preferred soil release agents are polymers of the generic formula:



in which each X can be a suitable capping group, with each X typically being selected from the group consisting of H, and alkyl or acyl groups containing from about 1 to about 4 carbon atoms. p is selected for water solubility and generally is from about 6 to about 113, preferably from about 20 to about 50. u is critical to formulation in a liquid composition having a relatively high ionic strength. There should be very little material in which u is greater than 10. Furthermore, there should be at least 20%, preferably at least 40%, of material in which u ranges from about 3 to about 5.

[0050] The R^{14} moieties are essentially 1,4-phenylene moieties. As used herein, the term "the R^{14} moieties are essentially 1,4-phenylene moieties" refers to compounds where the R^{14} moieties consist entirely of 1,4-phenylene moieties, or are partially substituted with other arylene or alkarylene moieties, alkylene moieties, alkenylene moieties, or mixtures thereof. Arylene and alkarylene moieties which can be partially substituted for 1,4-phenylene include 1,3-phenylene, 1,2-phenylene, 1,8-naphthylene, 1,4-naphthylene, 2,2-biphenylene, 4,4-biphenylene, and mixtures thereof. Alkylene and alkenylene moieties which can be partially substituted include 1,2-propylene, 1,4-butylene, 1,5-pentylene, 1,6-hexamethylene, 1,7-heptamethylene, 1,8-octamethylene, 1,4-cyclohexylene, and mixtures thereof.

[0051] For the R^{14} moieties, the degree of partial substitution with moieties other than 1,4-phenylene should be such that the soil release properties of the compound are not adversely affected to any great extent. Generally the degree of partial substitution which can be tolerated will depend upon the backbone length of the compound, i.e., longer backbones can have greater partial substitution for 1,4-phenylene moieties. Usually, compounds where the R^{14} comprise from about 50% to about 100% 1,4-phenylene moieties (from 0% to about 50% moieties other than 1,4-phenylene) have adequate soil release activity. For example, polyesters made according to the present invention with a 40:60 mole ratio of isophthalic (1,3-phenylene) to terephthalic (1,4-phenylene) acid have adequate soil release activity. However, because most polyesters used in fiber making comprise ethylene terephthalate units, it is usually desirable to minimize the degree of partial substitution with moieties other than 1,4-phenylene for best soil release activity. Preferably, the R^{14} moieties consist entirely of (i.e., comprise 100%) 1,4-phenylene moieties, i.e., each R^{14} moiety is 1,4-phenylene.

[0052] For the R^{15} moieties, suitable ethylene or substituted ethylene moieties include ethylene, 1,2-propylene, 1,2-butylene, 1,2-hexylene, 3-methoxy-1,2-propylene, and mixtures thereof. Preferably, the R^{15} moieties are essentially ethylene moieties, 1,2-propylene moieties, or mixtures thereof. Inclusion of a greater percentage of ethylene moieties tends to improve the soil release activity of compounds. Surprisingly, inclusion of a greater percentage of 1,2-propylene moieties tends to improve the water solubility of compounds.

[0053] Therefore, the use of 1,2-propylene moieties or a similar branched equivalent is desirable for incorporation of any substantial part of the soil release component in the liquid fabric softener compositions. Preferably, from about 75% to about 100%, are 1,2-propylene moieties.

[0054] The value for each p is at least about 6, and preferably is at least about 10. The value for each n usually ranges from about 12 to about 113. Typically the value for each p is in the range of from about 12 to about 43.

[0055] A more complete disclosure of soil release agents is contained in U.S. Pat. Nos.: 4,661,267, Decker, Konig, Straathof, and Gosselink, issued Apr. 28, 1987; 4,711,730, Gosselink and Diehl, issued Dec. 8, 1987; 4,749,596, Evans, Huntington, Stewart, Wolf and Zimmerer, issued June 7, 1988; 4,818,569, Trinh, Gosselink, and Rattinger, issued April 4, 1989; 4,877,896, Maldonado, Trinh, and Gosselink, issued Oct. 31, 1989; 4,956,447, Gosselink et al., issued Sept. 11, 1990; and 4,976,879, Maldonado, Trinh, and Gosselink, issued Dec. 11, 1990.

[0056] These soil release agents can also act as scum dispersants.

4. Scum Dispersant

[0057] In the present invention, the premix can be combined with an optional scum dispersant, other than the soil release agent, and heated to a temperature at or above the melting point(s) of the components.

[0058] The preferred scum dispersants herein are formed by highly ethoxylating hydrophobic materials. The hydro-

phobic material can be a fatty alcohol, fatty acid, fatty amine, fatty acid amide, amine oxide, quaternary ammonium compound, or the hydrophobic moieties used to form soil release polymers. The preferred scum dispersants are highly ethoxylated, e.g., more than about 17, preferably more than about 25, more preferably more than about 40, moles of ethylene oxide per molecule on the average, with the polyethylene oxide portion being from about 76% to about 97%, preferably from about 81% to about 94%, of the total molecular weight.

[0059] The level of scum dispersant is sufficient to keep the scum at an acceptable, preferably unnoticeable to the consumer, level under the conditions of use, but not enough to adversely affect softening. For some purposes it is desirable that the scum is nonexistent. Depending on the amount of anionic or nonionic detergent, etc., used in the wash cycle of a typical laundering process, the efficiency of the rinsing steps prior to the introduction of the compositions herein, and the water hardness, the amount of anionic or nonionic detergent surfactant and detergency builder (especially phosphates and zeolites) entrapped in the fabric (laundry) will vary. Normally, the minimum amount of scum dispersant should be used to avoid adversely affecting softening properties. Typically scum dispersion requires at least about 2%, preferably at least about 4% (at least 6% and preferably at least 10% for maximum scum avoidance) based upon the level of softener active. However, at levels of about 10% (relative to the softener material) or more, one risks loss of softening efficacy of the product especially when the fabrics contain high proportions of nonionic surfactant which has been absorbed during the washing operation.

[0060] Preferred scum dispersants are: Brij 700; Varonic U-250; Genapol T-500, Genapol T-800; Plurafac A-79; and Neodol 25-50.

5. Bactericides

[0061] Examples of bactericides used in the compositions of this invention include glutaraldehyde, formaldehyde, 2-bromo-2-nitro-propane-1,3-diol sold by Inolex Chemicals, located in Philadelphia, Pennsylvania, under trade name Bronopol, and a mixture of 5-chloro-2-methyl-4-isothiazoline-3-one and 2-methyl-4-isothiazoline-3-one sold by Rohm and Haas Company under the trade name Kathon CG/ICP. Typical levels of bactericides used in the present compositions are from about 1 to about 1,000 ppm by weight of the agent.

6. Other Optional Ingredients

[0062] The present invention can include optional components conventionally used in textile treatment compositions, for example, short chain alcohols such as ethanol, or propylene glycol, colorants, perfumes, preservatives, optical brighteners, opacifiers, surfactants, stabilizers such as guar gum and polyethylene glycol, anti-shrinkage agents, fabric crisping agents, spotting agents, germicides, fungicides, anti-oxidants such as butylated hydroxy toluene, anti-corrosion agents, and the like.

[0063] The compositions of the present invention are preferably used in the rinse cycle of the conventional automatic laundry operations. Generally, rinse water has a temperature of from about 15°C to about 60°C.

[0064] Fabrics or fibers are contacted with an effective amount, generally from about 20 ml to about 300 ml (per 3.5 kg of fiber or fabric being treated), of the compositions herein in an aqueous bath. Of course, the amount used is based upon the judgment of the user, depending on concentration of the softening materials, (A) + (B), fiber or fabric type, degree of softness desired, and the like. Typically, from about 20 ml to about 300 ml of 9% to 40% dispersion of the softening materials (A) + (B) are used in a 25 gallon laundry rinse bath to soften and provide antistatic benefits to a 3.5 kg load of mixed fabrics. Preferably, the rinse bath contains from about 20 ppm to about 250 ppm of the fabric softening materials (A) + (B) herein. More preferably for United States conditions, the rinse bath contains from about 50 ppm to about 150 ppm of the fabric softening materials (A) + (B). More preferably for European conditions, the rinse bath contains from about 250 ppm to about 450 ppm of the fabric softening materials (A) + (B). More preferably for Japanese conditions, the rinse bath contains from about 30 ppm to about 80 ppm of the fabric softening materials (A) + (B). These concentration levels achieve superior fabric softening and static control.

[0065] The invention is exemplified by the following non-limiting examples in which all numerical values are approximations consistent with normal experience.

EXAMPLE I					
FORMULA:	1	2	3	4	5
	(Wt.%)				
Di(2-amidoethyl) methyl quaternary ammonium salt ¹	0	6.25	12.50	18.75	25.00

¹Di(2-tallowamidoethyl)ethoxylated methyl ammonium methylsulfate (sold under the tradename Varisoft 222).

(continued)

EXAMPLE I					
FORMULA:	1	2	3	4	5
	(Wt.%)				
DEQA ²	24.5	18.75	12.50	6.25	0
Genapol® T-110	1.00	1.00	1.00	1.00	1.00
Ethanol	0.95	0.92	0.61	0.30	0.00
Isopropanol	0.00	0.68	1.39	2.08	2.78
Perfume	1.20	1.20	1.20	1.20	1.20
Silicone DC-200*	0.19	0.19	0.19	0.19	0.19
Silicone Antifoam* DC-2210	0.32	0.32	0.32	0.32	0.32
Soil Release Polymer	0.50	0.50	0.50	0.50	0.50
HCl	0.03	0.03	0.03	0.03	0.03
Lysine HCl	0.75	0.75	0.75	0.75	0.75
Deionized Water	70.56	69.40	69.01	68.63	68.23

* Product of the Dow-Corning Corporation.

²N,N-di(hydrogenated tallowyl-oxy-ethyl) N,N-dimethyl ammonium chloride

EXAMPLE I PREPARATION

[0066] The first three ingredients in each Formula are co-melted in a pyrex beaker, covered with a concave watch-glass, for three hours at about 80-85°C to form the premix. The water, HCl, and silicone antifoam are separately weighed into a sealed container and heated to about 83°C to form the water seat. The premix is then injected into the water seat over three to four minutes at about 72°C while stirring at from about 1500 to about 3000 r.p.m. A 15% aqueous solution of Lysine/HCl is added to the water seat/premix dispersion over seven minutes at about 71°C while stirring at from about 500 to about 1000 r.p.m.. The perfume, and the silicone DC-200 are added to the dispersion over thirty seconds. The dispersion is milled for two minutes at about 70°C at from about 4000 to about 8000 r.p.m. of the mill. A 40% aqueous solution of soil release polymer is added to the dispersion over two minutes at about 66°C while stirring at from about 500 to about 1000 r.p.m.. The dispersion is then chilled in an ice bath at about 25°C for 8 minutes while stirring at from about 200 to about 500 r.p.m..

Formula	Initial Viscosity (cP ³)	# Days	0°C (cP)	4°C (cP)	10°C (cP)
1	59	145	Gel(2d ⁴)	Gel(3d)	Gel(3d)
2	243	28	Gel(2d)	Gel(3d)	275
3	281	28	Gel(3d)	>500	248
4	143	28	Did not gel	133	132
5	155	28	Did not gel	>500	>500

³The abbreviation "cP" denotes centipoise.⁴The abbreviation "d" denotes days.

[0067] Concentrated compositions containing mixtures of the two softener active materials, Component (A) and Component (B), (Formulas 2-4), have more stable viscosities at temperatures down to 10°C, than compositions containing only the individual softener active Component (A) or Component (B), (Formulas 1 and 5).

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COMPARATIVE EXAMPLE II			
FORMULA	1	2	3
	(Wt.%)		
Ditallow Imidazoline Amide ⁵	25	----	16.67
DEQA ⁶	----	25	8.33
Soil Release Polymer	----	0.5	0.17
CaCl ₂	0.4	0.35	0.38
Perfume	1.35	1.35	1.35
DC-200 Silicone*	0.19	0.19	0.19
DC-2210 Silicone* Antifoam	0.32	0.32	0.32
Preservative(Kathon)	0.0003	0.0003	0.0003
HCl	1.70	0.02	1.14
EtOH	----	3.5	1.17
Deionized Water	71.0	68.8	70.3

* Product of the Dow-Corning Corporation.

⁵ 1-[hydrogenated tallowyl amido]ethyl-2-hydrogenated tallow imidazoline

⁶ N,N-di(hydrogenated tallowyl-oxy-ethyl)-N,N-dimethyl ammonium chloride

EXAMPLE II PREPARATION

Formula 1 Preparation

[0068] Ditallow Imidazoline Amide in the amount of 375g is melted by heating to about 93°C. This molten softener is then dispersed into 376g of deionized water at about 82°C containing 90g of a 28.25% HCl solution and 4.8g of the DC-2210 silicone antifoam over five minutes while stirring with an IKA model RW20 DZM stirrer at 400-600 r.p.m.. The resulting dispersion is stirred for an additional four minutes. A second aliquot of about 82°C deionized water in the amount of 580g is stirred into the thick dispersion over two and one half minutes. A blend of 20.25g of perfume and 2.85g of DC-200 silicone fluid is then added to the dispersion over thirty seconds, followed by two and one half minutes of mixing with an IKA Ultra-Turrax T50 high shear mill at 8000 r.p.m.. The dispersion is then cooled to room temperature over three minutes by passing it through a small plate and frame heat exchanger. The preservative in the amount of 0.3g is added at room temperature following the cool down.

Formula 2 Preparation

[0069] N,N-di(hydrogenated tallowyl-oxy-ethyl)-N,N-dimethyl ammonium chloride in the amount of 431g and 24g of ethanol are melted at about 91°C. This molten softener is dispersed into 954g of deionized water at about 82°C containing 8.25g of a 0.968 N HCl solution, 4.8g of DC-2210 silicone antifoam and 18.75g of a 40% solution of soil release polymer over five and one half minutes while stirring at 800-1100 r.p.m.. Subsequently, 35g of an aqueous 15% CaCl₂ solution is stirred into the dispersion over four and one half minutes. A blend of 20.25g of perfume and 2.85g of DC-200 silicone fluid is then added to the dispersion over thirty seconds, followed by two and one half minutes of mixing with a high shear mill at 8000 r.p.m.. The preservative in the amount of 0.3g is added just prior to cool-out. The dispersion is then cooled to room temperature over two and one half minutes by passing through a small plate and frame heat exchanger.

Formula 3 Preparation

[0070] An amount of 105g of Formula 2 is mixed with 210g of Formula 1 at room temperature.

Formula	Initial Viscosity (cP)	Days at 4°C	Viscosity after exposure to 4°C (cP)
1	38	5	>5000
2	78	3	gelled

(continued)

Formula	Initial Viscosity (cP)	Days at 4°C	Viscosity after exposure to 4°C (cP)
3	43	3	gelled

[0071] The above table demonstrates the disadvantage of the preparation process in which the actives are not co-melted as described in Example II. The use of a molten premix of the softener actives Component (A) and Component (B), provides a product with superior viscosity stability.

EXAMPLE (Not within the scope of the invention)		III	IV	V	VI
		(Wt.%)			
Ditallow Imidazoline Amide ⁷		14.3	14.3	14.3	11.0
DEQA ⁸		7.7	8.7	9.7	12.0
CaCl ₂		0.375	0.375	0.375	0.375
Perfume		1.35	1.35	1.35	1.35
DC-200 Silicone*		0.19	0.19	0.19	0.19
DC-2210 Silicone*		0.32	0.32	0.32	0.32
Preservative(Kathon) Antifoam		0.0003	0.0003	0.003	0.003
HCl		0.97	0.97	0.97	0.75
EtOH		1.25	1.42	1.58	1.95
Deionized Water		73.5	72.3	71.2	72.1

* Product of the Dow-Corning Corporation.

⁷ 1-[hydrogenated tallowyl amido]ethyl-2-hydrogenated tallow imidazoline

⁸ N,N-di(hydrogenated tallowyl-oxy-ethyl)-N,N-dimethyl ammonium chloride

EXAMPLE PREPARATION

Example III

[0072] Ditallow Imidazoline Amide in the amount of 214g, 115.5g N,N-di(tallowyl-oxy-ethyl)-N,N-dimethyl ammonium chloride and 19g of ethanol are co-melted at about 91 °C. This molten softener mixture is dispersed into 1,102g of deionized water at about 82°C containing 14.55g of HCl and 4.8g of DC-2210 silicone antifoam over five and one half minutes while stirring at 800-1100 r.p.m.. Subsequently, 37.5g of an aqueous 15% CaCl₂ solution is stirred into the dispersion over four and one half minutes. A blend of 20.25g of perfume and 2.85g of DC-200 silicone fluid is then added to the dispersion over thirty seconds, followed by two and one half minutes of mixing with high shear mill at 8000 r.p.m.. The dispersion is then cooled to room temperature over two and one half minutes by passing it through a small plate and frame heat exchanger.

[0073] The preservative in the amount of 0.3g is added at room temperature following cool-out.

Examples IV, V and VI

[0074] These Examples are prepared in the same manner as Example III except that the amounts of imidazoline, N,N-di(hydrogenated tallowyl-oxy-ethyl)-N,N-dimethyl ammonium chloride, ethanol, and HCl vary according to the formulas shown above.

Example	Initial Viscosity (cP)	Days at 4°C	Viscosity after exposure to 4°C (cP)
III	18	42	60
IV	15	23	50
V	18	30	95
VI	18	29	33

[0075] This demonstrates the advantage of using a molten premix and the type of high shear dispersion that is possible with a high shear mill.

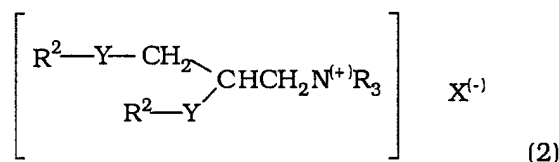
Claims

1. A stable, aqueous, liquid, concentrated fabric softening composition comprising:

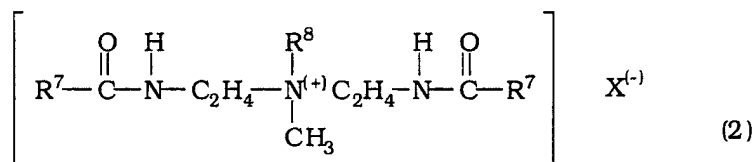
(A) biodegradable diester quaternary ammonium fabric softening material selected from the group consisting of:



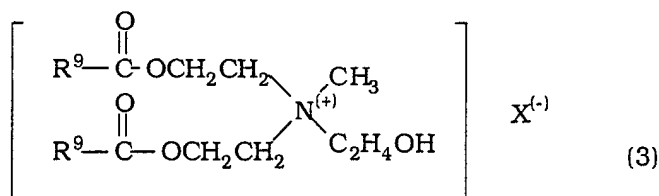
wherein each R is selected from the group consisting of C₁ to C₆ alkyl groups; each m is 2, each n is from 1 to 4; each R² is selected from the group consisting of from C₁₂ to C₂₂ hydrocarbyl or substituted hydrocarbyl group such that the Iodine Value (IV) of the parent fatty acid of this R² group is less than 10; each Y is -O-(O)C- or -C(O)-O-, but not -OC(O)O-; and wherein X⁽⁻⁾ is a compatible anion; or



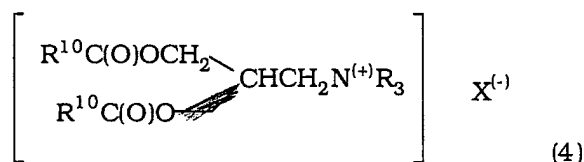
wherein each Y, R² and X⁽⁻⁾ have the same meanings as before; or mixtures thereof; and
(B) co-active fabric softening material selected from the group consisting of:



wherein each R⁷ is selected from the group consisting of C₁₄ to C₂₀ alkyl and alkenyl groups; each R⁸ is selected from methyl, ethyl, and -(C_mH_{2m}O)_nH; wherein n³ is from 1 to 5; and wherein each m, and X⁽⁻⁾ have the same meaning as before; or



wherein each R⁹ is a C₁₅-C₁₇ fatty alkyl group such that the Iodine Value (IV) of the parent fatty acid of this R⁹ group is from 20 to 100, and X⁽⁻⁾ has the same meaning as before; or



wherein each R^{10} is a C_{12} - C_{22} hydrocarbyl or substituted hydrocarbyl substituent, such that the Iodine Value (IV) of the parent fatty acid of this R^{10} group is from 20 to 100, and R and $\text{X}^{(-)}$ have the same meaning as before; or

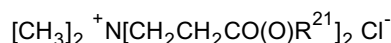
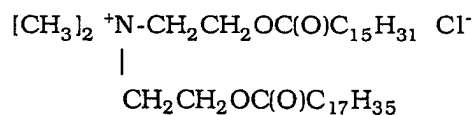
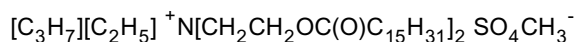
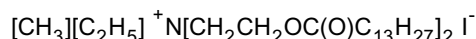
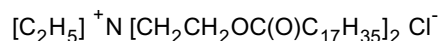
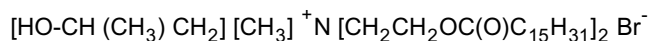


except for those according to (3), wherein each R^{11} is a long chain C_{12} - C_{22} hydrocarbyl, or substituted hydrocarbyl substituent, such that the Iodine Value (IV) of the parent fatty acid of this R^{11} group is from 20 to 100, and each m, n, Y, R and $\text{X}^{(-)}$ have the same meaning as before; or mixtures thereof; wherein the total amount of components (A) + (B) is from 15% to 35%, preferably from 20% to 32%, more preferably from 22% to 27%, wherein the ratio of (A) to (B) is from 0.25:1 to 4:1, and even more preferably from 0.3:1 to 1.5:1; wherein when (A) is (1), (B) is not (5); wherein when (A) is (2), (B) is not (4); wherein when (A) is (1) with one R being methyl and the other being $\text{C}_2\text{H}_4\text{OH}$, (B) is not (3); wherein when B (3) is $[\text{CH}_2\text{CH}_2\text{OH}][\text{CH}_3]^+\text{N}[(\text{CH}_2\text{CH}_2\text{OC(O)R}^2)]_2\text{X}^{(-)}$, $\text{X}^{(-)}$ being as hereinbefore defined, A is not (1);

wherein when B (3) is



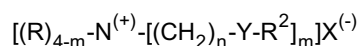
where $-\text{C(O)R}^{20}$ is derived from partially hydrogenated tallow or modified tallow, $\text{X}^{(-)}$ being as hereinbefore defined, A is not



wherein $-\text{CO(O)R}^{21}$ is derived from saturated tallow; and preferably wherein the final aqueous concentrated fabric softening agent has a neat pH of from 2.5 to 4.

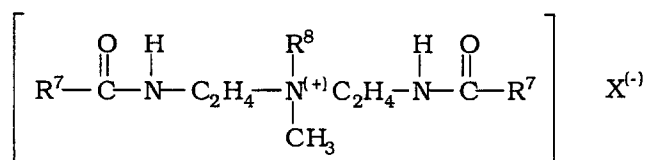
2. The stable, aqueous, liquid, concentrated fabric softening composition according to Claim 1 comprising:

(A) biodegradable diester quaternary ammonium fabric softening material having the formula:



wherein each R is selected from the group consisting of C₁ to C₆ alkyl groups; each m is 2; each n is from 1 to 4; each R² is selected from the group consisting of C₁₂ to C₂₂ hydrocarbyl or substituted hydrocarbyl group, such that the IV of the parent fatty acid of this R² group is less than 10, each Y is -O-(O)C- or -C(O)-O-, but not -OC(O)O; and wherein X⁽⁻⁾ is a compatible anion; or

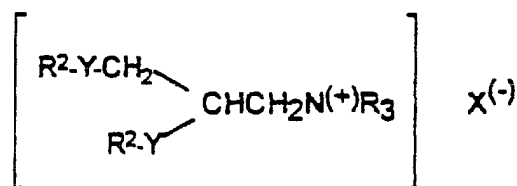
(B) co-active fabric softening material having the formula:



wherein each R⁷ is selected from the group consisting of C₁₄ to C₂₀ alkyl and alkenyl groups; each R⁸ is selected from methyl, ethyl, and -(C_mH_{2m}O)_n3H, wherein n³ is from 1 to 5; and wherein each m, and X⁽⁻⁾ have the same meaning as before.

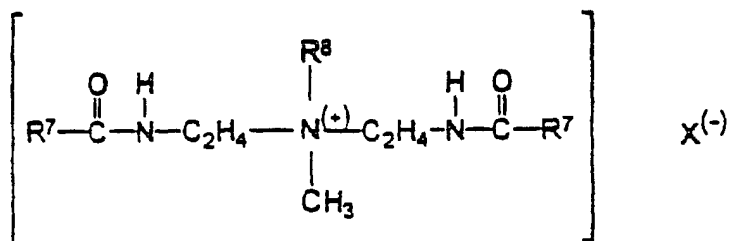
3. The stable, aqueous, liquid, concentrated fabric softening composition according to Claim 1 comprising:

(A) biodegradable diester quaternary ammonium fabric softening material having the formula:



wherein each Y is -O(O)C, or -C(O)O-, but not -OC(O)O; and wherein each R is selected from the group consisting of C₁ to C₆ alkyl, or hydroxyalkyl, groups, each R² is selected from the group consisting of from C₁₂ to C₂₂ hydrocarbyl or substituted hydrocarbyl group such that the of the parent fatty acid of this R² group is less than 10, and wherein X⁽⁻⁾ is a compatible anion; and

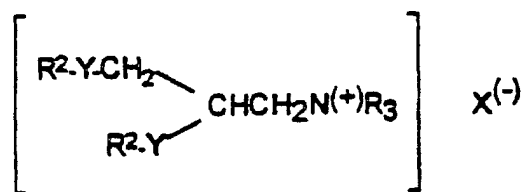
(B) co-active fabric softening material having the formula:



wherein each R⁷ is selected from the group consisting of C₁₄ to C₂₀ alkyl and alkenyl groups; each R⁸ is selected from methyl, ethyl, and -(C_mH_{2m}O)_n3H, wherein n³ is from 1 to 5; and wherein each m, and X⁽⁻⁾ have the same meaning as before.

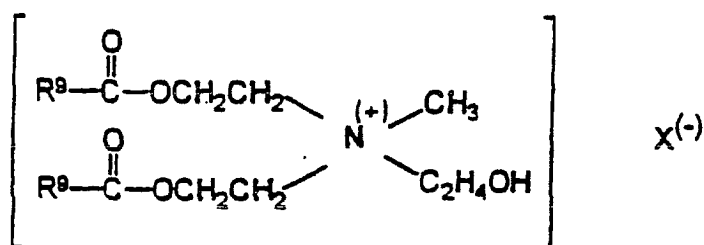
4. The stable, aqueous, liquid, concentrated fabric softening composition according to Claim 1 comprising:

(A) biodegradable diester quaternary ammonium fabric softening material having the formula:



wherein each Y is -O(O)G, or -C(O)O-, but not -OC(O)O; and wherein each R is selected from the group consisting of C₁ to C₆ alkyl, or hydroxyalkyl, groups, each R² is selected from the group consisting of from C₁₂ to C₂₂ hydrocarbyl or substituted hydrocarbyl group such that the of the parent fatty acid of this R² group is less than 10, and wherein X⁽⁻⁾ is a compatible anion; and

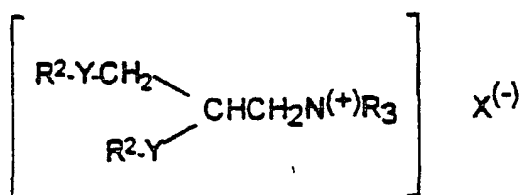
(B) co-active fabric softening material having the formula:



wherein each R⁹ is a C₁₅-C₁₇ fatty alkyl group such that the IV of the parent fatty acid of this R⁹ group is from 20 to 100, and X⁽⁻⁾ has the same meaning as before.

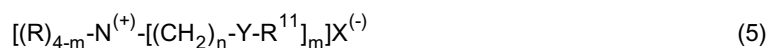
5. The stable, aqueous, liquid, concentrated fabric softening composition according to Claim 1 comprising:

(A) biodegradable diester quaternary ammonium fabric softening material having the formula:



wherein each Y is -O(O)C, or -C(O)O-, but not -OC(O)O; and wherein each R is selected from the group consisting of C₁ to C₆ alkyl, or hydroxyalkyl, groups, each R² is selected from the group consisting of from C₁₂ to C₂₂ hydrocarbyl or substituted hydrocarbyl group such that the of the parent fatty acid of this R² group is less than 10, and wherein X⁽⁻⁾ is a compatible anion; and

(B) co-active fabric softening material having the formula:



wherein each each R¹¹ is a long chain C₁₂-C₂₂ hydrocarbyl, or substituted hydrocarbyl substituent, such that the of the parent fatty acid of this R¹¹ group is from 20 to 100, and each m, n, Y, R and X⁽⁻⁾ have the same meaning as before or mixtures thereof.

6. A process for making a composition as defined in any one of Claims 1-5, which comprises the steps of using a molten premix comprising:

A. premixing (A) biodegradable diester quaternary ammonium fabric softening agent, and (B) secondary fabric softening agent; and

B. injecting the premix into an acid/water seat preferably comprising acid selected from the group consisting of the inorganic mineral acids and the organic acids having the formula R¹²COOH, wherein R¹² is a C₁-C₅ H or alkyl group or R¹³CH₂SO₃H, wherein R¹³ is hydrogen or C₁ to C₄ alkyl, and mixtures thereof; more preferably - wherein the acid is phosphoric acid, hydrochloric acid, citric acid or mixtures thereof; preferably wherein the acid concentration of the acid/water seat is up to 2%; and preferably wherein the acid to (B) molar ratio is from 0:1 to 1.2:1; and

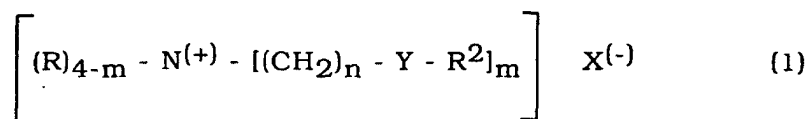
C. conducting high shear milling and adding electrolyte preferably selected from the group consisting of salts of Group IA and IIA metals of the periodic table of elements, preferably calcium chloride or ammonium salts.

7. The composition according to Claim 8 wherein said premix is combined with a scum dispersant.

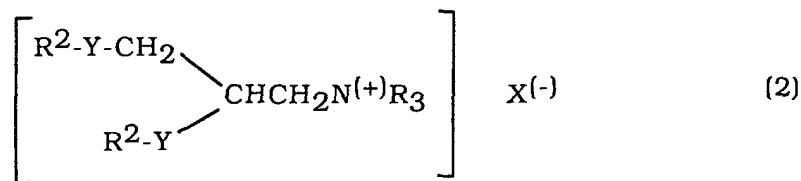
Patentansprüche

1. Stabile, wäßrige, flüssige, konzentrierte Wäscheweichmachierzusammensetzung, umfassend:

(A) bioabbaubares quaternäres Diester-Ammonium-Wäscheweichmachermaterial, gewählt aus der Gruppe, bestehend aus:

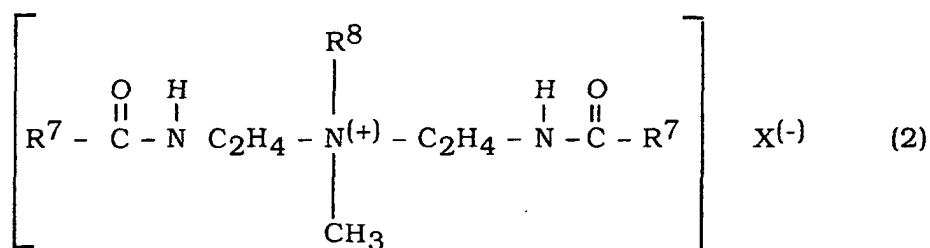


worin jedes R aus der Gruppe gewählt ist, bestehend aus C₁-C₆-Alkylgruppen; jedes m 2 ist, jedes n 1 bis 4 ist; jedes R² aus der Gruppe gewählt ist, bestehend aus C₁₂-C₂₂-Hydrocarbyl- oder substituierten Hydrocarbylgruppen, so daß die Iodzahl (IV) der Stammfettsäure dieser R²-Gruppe weniger als 10 beträgt; jedes Y die Bedeutung -O-(O)C- oder -C(O)-O-, jedoch nicht OC(O)O hat; und wobei X⁽⁻⁾ ein kompatibles Anion ist; oder

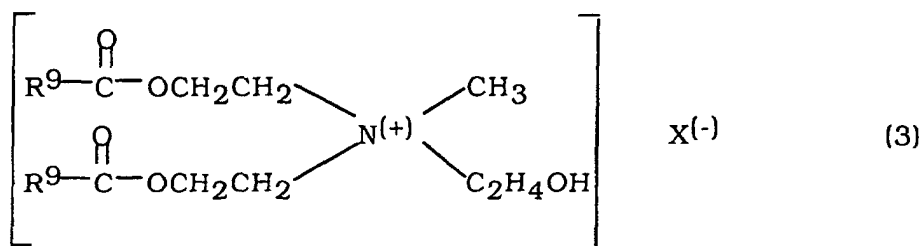


worin Y, R, R² und X⁽⁻⁾ jeweils die gleichen Bedeutungen wie oben haben; oder Mischungen hiervon; und

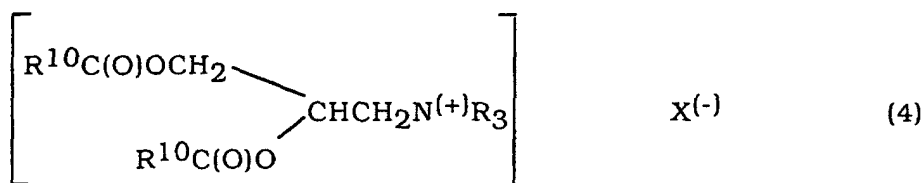
(B) coaktives Wäscheweichmachermaterial, gewählt aus der Gruppe, bestehend aus:



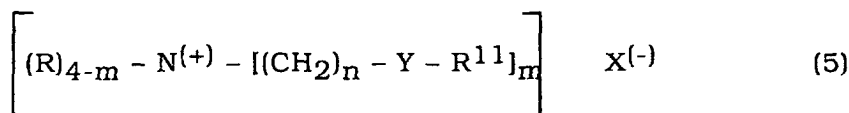
worin jedes R⁷ aus der Gruppe gewählt ist, bestehend aus C₁₄-C₂₀-Alkyl- und -Alkenylgruppen; jedes R⁸ aus Methyl, Ethyl und -(C_mH_{2m}O)_n3H gewählt ist; worin n³ 1 bis 5 ist; und worin m und X⁽⁻⁾ jeweils die gleichen Bedeutungen wie oben haben; oder



worin jedes R⁹ eine C₁₅-C₁₇-Fettalkylgruppe ist, so daß die Iodzahl (IV) der Stammfettsäure dieser R⁹-Gruppe 20 bis 100 beträgt, und X⁽⁻⁾ die gleiche Bedeutung wie oben hat; oder



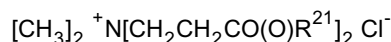
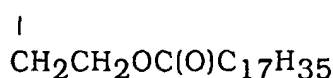
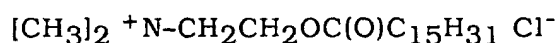
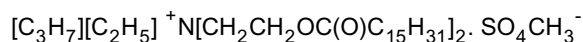
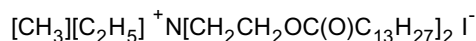
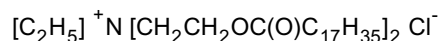
worin jedes R¹⁰ ein C₁₂-C₂₂-Hydrocarbyl- oder substituierter Hydrocarbylsubstituent ist, so daß die Iodzahl (IV) der Stammfettsäure dieser R¹⁰-Gruppe 20 bis 100 beträgt, und R und X⁽⁻⁾ die gleichen Bedeutungen wie oben haben; oder



mit Ausnahme derjenigen gemäß (3), worin jedes R¹¹ ein langkettiger C₁₂-C₂₂-Hydrocarbyl- oder substituierter Hydrocarbylsubstituent ist, so daß die Iodzahl (IV) der Stammfettsäure dieser R¹¹-Gruppe 20 bis 100 beträgt, und m, n, Y, R und X⁽⁻⁾ jeweils die gleichen Bedeutungen wie oben haben; oder Mischungen hiervon; wobei die Gesamtmenge der Komponenten (A) + (B) 15 bis 35%, vorzugsweise 20 bis 32%, weiter vorzugsweise 22 bis 27%, beträgt, wobei das Verhältnis von (A) zu (B) 0,25: 1 bis 4:1, und noch weiter bevorzugt 0,3:1 bis 1,5:1 beträgt; wobei, wenn (A) die Bedeutung (1) hat, (B) nicht (5) ist; wobei, wenn (A) die Bedeutung (2) hat, (B) nicht (4) ist; wobei, wenn (A) die Bedeutung (1) hat, wobei ein R Methyl und das andere C₂H₄OH ist, (B) nicht (3) ist; wobei, wenn B (3)

[CH₂CH₂OH] [CH₃] + N [(CH₂CH₂OC(O) R₂] X⁽⁻⁾ ist, X- wie vorangehend definiert ist, A nicht (1) ist; wobei, wenn B (3)

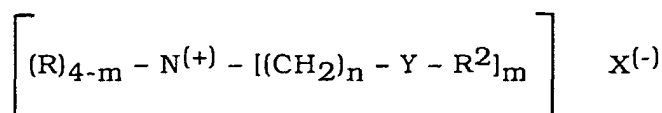
$[\text{CH}_2\text{CH}_2\text{OH}][\text{CH}_3]^+\text{N}[(\text{CH}_2\text{CH}_2\text{OC}(\text{O})\text{R}_{20})_2]\text{Cl}^-$, worin $-\text{C}(\text{O})\text{R}_2$ von teilweise hydriertem Talg oder modifiziertem Talg abgeleitet ist, bedeutet und X- die oben angegebenen Bedeutungen besitzt, A nicht



worin $-\text{CO}(\text{O})\text{R}^{21}$ von gesättigtem Talg abgeleitet ist, ist;
und wobei vorzugsweise das fertige, wäßrige, konzentrierte Wäscheweichmachermittel einen unverdünnten pH von 2,5 bis 4 aufweist.

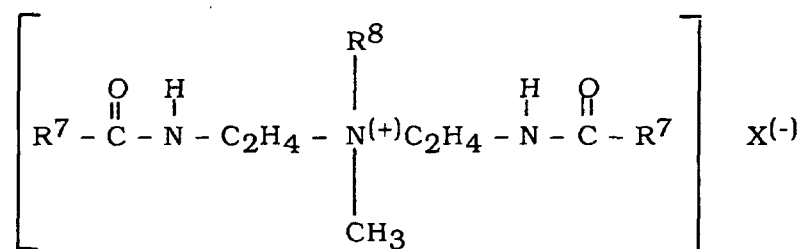
2. Stabile, wäßrige, flüssige, konzentrierte Wäscheweichmacherezusammensetzung nach Anspruch 1, umfassend:

(A) bioabbaubares, quaternäres Diester-Ammonium-Wäscheweichmachermaterial der Formel:



worin jedes R aus der Gruppe gewählt ist, bestehend aus C_1 - C_6 -Alkylgruppen; jedes m 2 ist; jedes n 1 bis 4 ist; jedes R^2 aus der Gruppe gewählt ist, bestehend aus C_{12} - C_{22} -Hydrocarbyl- oder substituierten Hydrocarbylgruppen, so daß die Iodzahl (IV) der Stammfettsäure dieser R^2 -Gruppe weniger als 10 beträgt; jedes Y -O-(O)C- oder -C(O)-O, jedoch nicht OC(O)O ist; und wobei $\text{X}^{(-)}$ ein kompatibles Anion ist; und

(B) coaktives Wäscheweichmachermaterial der Formel

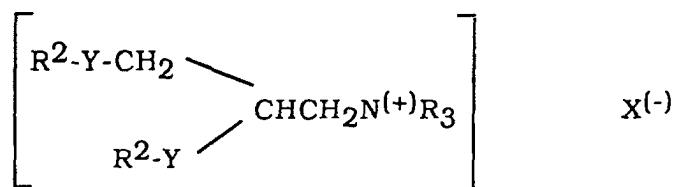


worin jedes R^7 aus der Gruppe gewählt ist, bestehend aus C_{14} - C_{20} -Alkyl- und -Alkenylgruppen; jedes R^8 aus Methyl, Ethyl und $-(\text{C}_m\text{H}_{2m}\text{O})_n\text{H}$ gewählt ist; worin n³ 1 bis 5 ist; und worin m und $\text{X}^{(-)}$ jeweils die gleiche Bedeutung

wie oben haben.

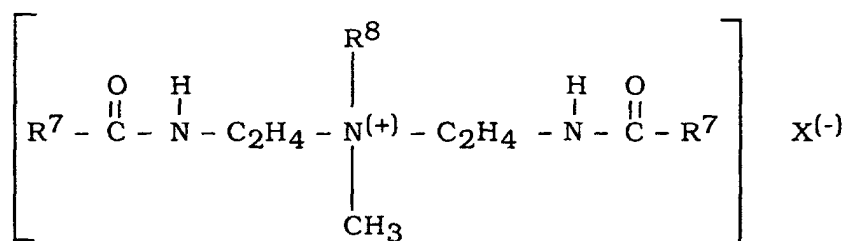
3. Stabile, wäßrige, flüssige, konzentrierte Wäscheweichmacherzusammensetzung nach Anspruch 1, umfassend

(A) bioabbaubares, quaternäres Diester-Ammonium-Wäscheweichmachermaterial der Formel:



worin jedes Y -O-(O)C oder -C(O)-O-, jedoch nicht -OC(O)O ist; und wobei jedes R aus der Gruppe gewählt ist, bestehend aus C₁-C₆-Alkyl- oder Hydroxyalkylgruppen; jedes R² aus der Gruppe gewählt ist, bestehend aus C₁₂-C₂₂-Hydrocarbyl- oder substituierten Hydrocarbylgruppen, so daß die Iodzahl (IV) der Stammfettsäure dieser R²-Gruppe weniger als 10 beträgt, und wobei X⁽⁻⁾ ein kompatibles Anion ist; und

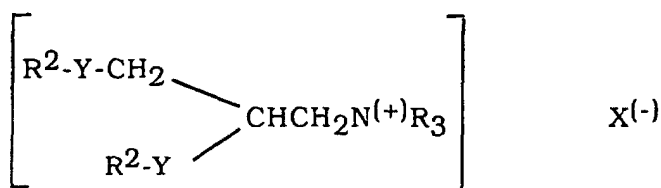
(B) coaktives Wäscheweichmachermaterial der Formel



worin jedes R⁷ aus der Gruppe gewählt ist, bestehend aus C₁₄-C₂₀-Alkyl- und -Alkenylgruppen; jedes R⁸ aus Methyl, Ethyl und -(C_mH_{2m}O)_n3H gewählt ist; worin n³ 1 bis 5 ist; und worin m und X⁽⁻⁾ jeweils die gleichen Bedeutungen wie oben haben.

4. Stabile, wäßrige, flüssige, konzentrierte Wäscheweichmacherzusammensetzung nach Anspruch 1, umfassend

(A) bioabbaubares, quaternäres Diester-Ammonium-Wäscheweichmachermaterial der Formel:

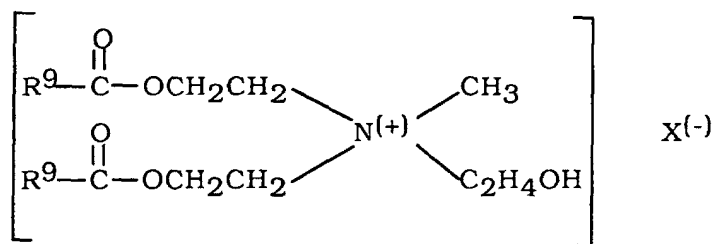


worin jedes Y -O(O)C oder -C(O)O-, jedoch nicht -OC(O)O ist; und worin jedes R aus der Gruppe gewählt ist, bestehend aus C₁-C₆-Alkyl- oder Hydroxyalkylgruppen; jedes R² aus der Gruppe gewählt ist, bestehend aus C₁₂-C₂₂-Hydrocarbyl- oder substituierten Hydrocarbylgruppen, so daß die Iodzahl (IV) der Stammfettsäure dieser R²-Gruppe weniger als 10 beträgt, und worin X⁽⁻⁾ ein kompatibles Anion ist; und

(B) coaktives Wäscheweichmachermaterial der Formel

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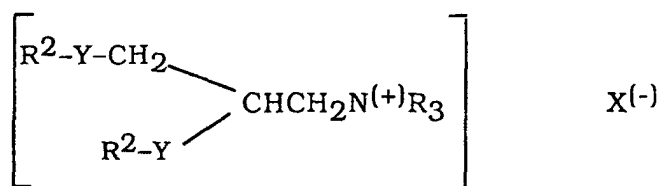
worin jedes R^9 eine C_{15} - C_{17} -Fettalkylgruppe ist, so daß die IV der Stammfettsäure dieser R^9 -Gruppe 20 bis 100 beträgt, und worin $\text{X}^{(-)}$ die gleiche Bedeutung wie oben hat.

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5. Stabile, wäßrige, flüssige, konzentrierte Wäscheweichmacherzusammensetzung nach Anspruch 1, umfassend

(A) bioabbaubares, quaternäres Diester-Ammonium-Wäscheweichmachermaterial der Formel:

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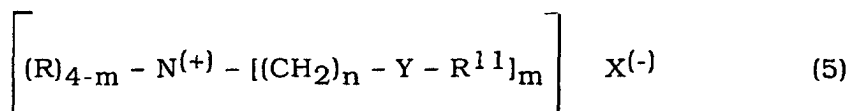
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worin jedes Y -O(O)C oder -C(O)O-, jedoch nicht -OC(O)O ist; und worin jedes R aus der Gruppe gewählt ist, bestehend aus C_1 - C_6 -Alkyl- oder -Hydroxyalkylgruppen; jedes R^2 aus der Gruppe gewählt ist, bestehend aus C_{12} - C_{22} -Hydrocarbyl- oder substituierten Hydrocarbylgruppen, so daß die Iodzahl (IV) der Stammfettsäure dieser R^2 -Gruppe weniger als 10 beträgt, und worin $\text{X}^{(-)}$ ein kompatibles Anion ist; und

(B) coaktives Wäscheweichmachermaterial der Formel

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worin jedes R^{11} ein langkettiger C_{12} - C_{22} -Hydrocarbyl- oder substituiertes Hydrocarbylsubstituent ist, so daß die Iodzahl (IV) der Stammfettsäure dieser R^{11} -Gruppe 20 bis 100 beträgt, und m, n, Y, R und $\text{X}^{(-)}$ jeweils die gleichen Bedeutungen wie oben haben; oder Mischungen hiervon.

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6. Verfahren zur Herstellung einer Zusammensetzung nach mindestens einem der Ansprüche 1-5, umfassend die Schritte der Verwendung einer geschmolzenen Vormischung, umfassend:

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A. Vormischen von (A) bioabbaubarem, quaternärem Diester-Ammonium-Wäscheweichmachermittel und (B) sekundärem Wäscheweichmachermittel; und

B. Injizieren der Vormischung in eine Säure/Wasser-Auflage, vorzugsweise umfassend eine Säure, gewählt aus der Gruppe, bestehend aus anorganischen Mineralsäuren und organischen Säuren der Formel R^{12}COOH , worin R^{12} eine C_1 - C_5 -H- oder Alkylgruppe ist, oder $\text{R}^{13} \text{CH}_2\text{SO}_3\text{H}$, worin R^{13} Wasserstoff oder C_1 - C_4 -Alkyl ist, und Mischungen hiervon; wobei weiter vorzugsweise die Säure Phosphorsäure, Chlorwasserstoffsäure, Citronensäure oder Mischungen hiervon ist; wobei vorzugsweise die Säurekonzentration der Säure/Wasser-Auflage bis zu 2% beträgt; und wobei vorzugsweise das Molverhältnis von Säure zu (B) 0:1 bis 1,2:1 beträgt; und

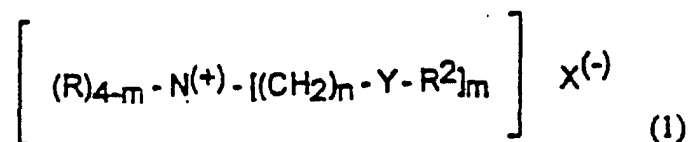
C. Durchführen von Hochscher-Mahlen und Zugeben von Elektrolyt, vorzugsweise gewählt aus der Gruppe, bestehend aus Salzen von Metallen der Gruppe IA und IIA des Periodensystems, vorzugsweise Calciumchlorid oder Ammoniumsalzen.

7. Verfahren nach Anspruch 6, wobei die Vormischung mit einem Schaumdispergiermittel kombiniert wird.

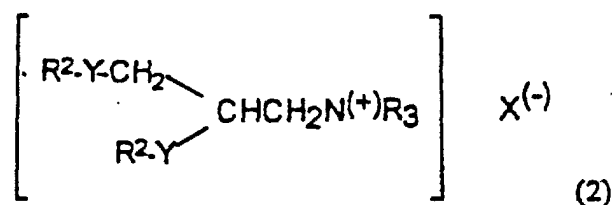
Revendications

1. Composition d'adoucissement de tissus liquide aqueuse concentrée stable comprenant :

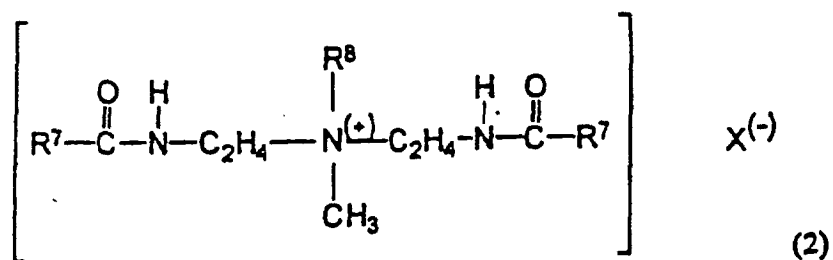
(A) un matériau adoucissant de tissus d'ammonium quaternaire biodégradable de type diester choisi dans le groupe constitué des substances de formule suivante :



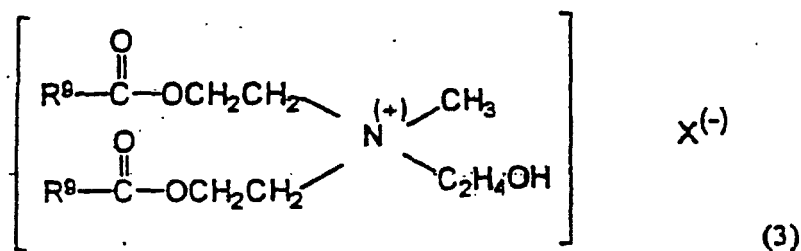
dans laquelle chaque groupe R est choisi dans le groupe constitué de groupes alkyle en C_1-C_6 ; chaque indice m est égal à 2 ; chaque indice n a une valeur de 1 à 4 ; chaque groupe R^2 est choisi dans le groupe constitué d'un groupe hydrocarbyle ou hydrocarbyle substitué en $C_{12}-C_{22}$ tel que l'indice d'iode (IV) de l'acide gras apparenté de ce groupe R^2 soit inférieur à 10 ; chaque groupe Y est un groupe $-O-(O)C$ ou $-C(O)-O$, mais pas $OC(O)O$; et dans laquelle $X^{(-)}$ est un anion compatible ; ou de formule :



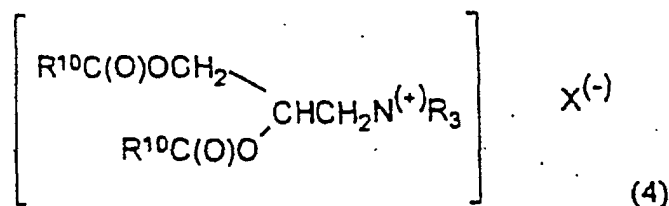
dans laquelle chaque groupe Y, R, R^2 et $X^{(-)}$ a la même signification que ci-dessus ; ou de leurs mélanges, et
(B) un matériau adoucissant de tissus co-actif choisi dans le groupe constitué des substances de formule :



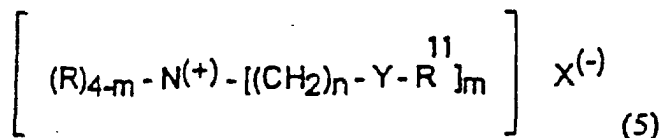
dans laquelle chaque groupe R^7 est choisi dans le groupe constitué des groupes alkyle et alcényle en $C_{14}-C_{20}$; chaque groupe R^8 est choisi parmi les groupes méthyle, éthyle et $-(C_mH_{2m}O)_n3H$; dans laquelle n^3 a une valeur de 1 à 5 ; et dans laquelle chaque indice m et le groupe $X^{(-)}$ ont la même signification que ci-dessus ; ou de formule :



dans laquelle chaque groupe R^9 est un groupe alkyle gras en C_{15} - C_{17} tel que l'indice d'iode (IV) de l'acide gras apparenté de ce groupe R^9 soit de 20 à 100; et $\text{X}^{(-)}$ a la même signification que ci-dessus; où de formule :



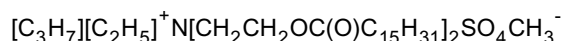
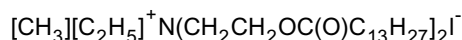
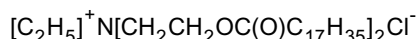
dans laquelle chaque groupe R^{10} est un substituant hydrocarbyle ou hydrocarbyle substitué en C_{12} - C_{22} tel que l'indice d'iode (IV) de l'acide gras apparenté de ce groupe R^{10} soit de 20 à 100; et R et $\text{X}^{(-)}$ ont la même signification que ci-dessus; ou de formule :

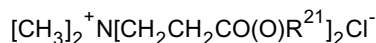
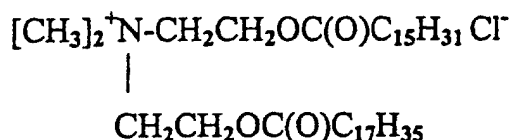


excepté pour les substances selon (3), dans laquelle chaque groupe R^{11} est un substituant hydrocarbyle ou hydrocarbyle substitué à longue chaîne en C_{12} - C_{22} ou tel que l'indice d'iode (IV) de l'acide gras apparenté de ce groupe R^{11} soit de 20 à 100, et chacun de m , n , Y , R et

$\text{X}^{(-)}$ a la même signification que ci-dessus; ou leurs mélanges; dans laquelle la quantité totale de composants (A) + (B) est de 15% à 35%, de préférence de 20% à 32%, mieux encore de 22% à 27%, le rapport de (A) à (B) étant de 0,25:1 à 4:1, et bien mieux encore de 0,3:1 à 1,5:1; dans laquelle, lorsque (A) est (1), (B) n'est pas (5); dans laquelle, lorsque (A) est (2), (B) n'est pas (4); dans laquelle, lorsque (A) est (1), un groupe R étant un groupe méthyle et l'autre étant un groupe $\text{C}_2\text{H}_4\text{OH}$, (B) n'est pas (3); dans laquelle, lorsque B(3) est $[\text{CH}_2\text{CH}_2\text{OH}]$ $[\text{CH}_3]^+\text{N}[(\text{CH}_2\text{CH}_2\text{OC})(\text{O})\text{R}^2]\text{X}^-$ où $\text{X}^{(-)}$ étant tel que défini ci-dessus, A n'est pas (1); dans laquelle lorsque B(3) est $[\text{CH}_2\text{CH}_2\text{OH}][\text{CH}_3]^+\text{N}[\text{CH}_2\text{CH}_2\text{OC}(\text{O})\text{R}^{20}]_2\text{Cl}^-$ où $-\text{C}(\text{O})\text{R}^{20}$ est dérivé de suif modifié ou de suif partiellement hydrogéné

X^- étant comme défini ci-dessus, A n'est pas

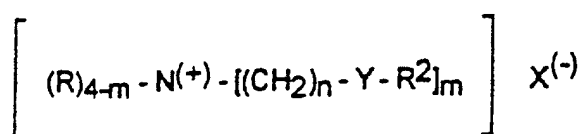




où $-\text{CO}(\text{O})\text{R}^{21}$ est dérivé de suif saturé et de préférence dans laquelle l'agent adoucissant de tissus aqueux concentré final a un pH à l'état non dilué de 2,5 à 4.

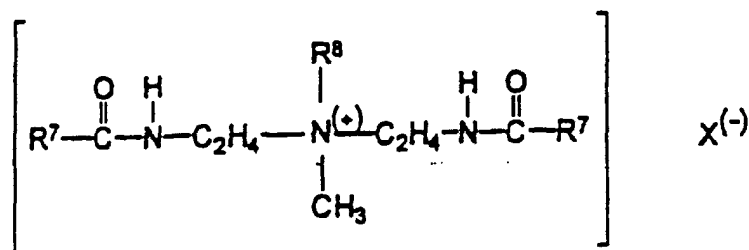
2. Composition d'adoucissement de tissus liquide aqueuse concentrée stable selon la revendication 1, comprenant :

(A) un matériau adoucissant de tissus d'ammonium quaternaire biodégradable de type diester ayant pour formule :



dans laquelle chaque groupe R est choisi dans le groupe constitué de groupes alkyle en $\text{C}_1\text{-C}_6$; chaque indice m est égal à 2 ; chaque indice n a une valeur de 1 à 4 ; chaque groupe R^2 est choisi dans le groupe constitué d'un groupe hydrocarbyle ou hydrocarbyle substitué en $\text{C}_{12}\text{-C}_{22}$ tel que le (IV) de l'acide gras apparenté de ce groupe R^2 soit inférieur à 10 ; chaque groupe Y est un groupe $-\text{O}(\text{O})\text{C}$ ou $-\text{C}(\text{O})\text{-O}$, mais pas $\text{OC}(\text{O})\text{O}$; et dans laquelle $\text{X}^{(-)}$ est un anion compatible ; et

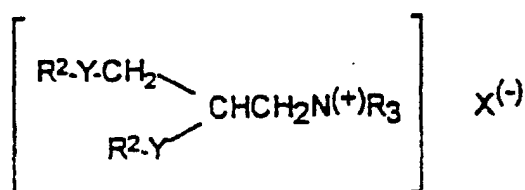
(B) un matériau adoucissant de tissus co-actif ayant pour formule :



dans laquelle chaque groupe R^7 est choisi dans le groupe constitué des groupes alkyle et alcényle en $\text{C}_{14}\text{-C}_{20}$; chaque groupe R^8 est choisi parmi les groupes méthyle, éthyle et $-(\text{C}_m\text{H}_{2m}\text{O})_n\text{3H}$; dans laquelle n^3 a une valeur de 1 à 5 ; et dans laquelle chaque indice m et le groupe $\text{X}^{(-)}$ ont la même signification que ci-dessus.

3. Composition d'adoucissement de tissus liquide aqueuse concentrée stable selon la revendication 1, comprenant :

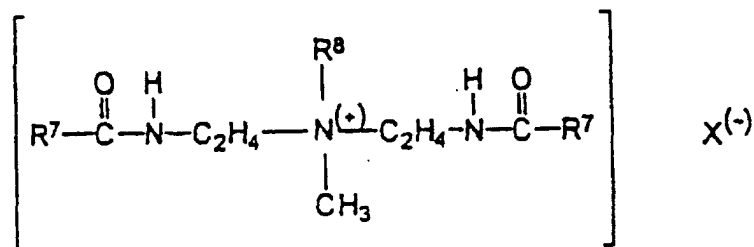
(A) un matériau adoucissant de tissus d'ammonium quaternaire biodégradable de type diester ayant pour formule :



dans laquelle chaque groupe Y est $-\text{O}(\text{O})\text{C}$ ou $-\text{C}(\text{O})\text{O-}$, mais pas $-\text{OC}(\text{O})\text{O-}$; et dans laquelle chaque

groupe R est choisi dans le groupe constitué de groupes alkyle ou hydroxyalkyle en C₁-C₆; chaque groupe R² est choisi dans le groupe constitué d'un groupe hydrocarbyle ou hydrocarbyle substitué en C₁₂-C₂₂ tel que le (IV) de l'acide gras apparenté de ce groupe R² soit inférieur à 10; et dans lequel X⁽⁻⁾ est un anion compatible; et

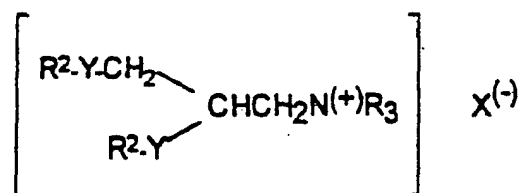
(B) un matériau adoucissant de tissus co-actif ayant pour formule :



dans laquelle chaque groupe R⁷ est choisi dans le groupe constitué des groupes alkyle et alcényle en C₁₄-C₂₀; chaque groupe R⁸ est choisi parmi les groupes méthyle, éthyle et —(C_mH_{2m}O)_n3H; dans laquelle n³ a une valeur de 1 à 5; et dans laquelle chaque indice m et le groupe X⁽⁻⁾ ont la même signification que ci-dessus.

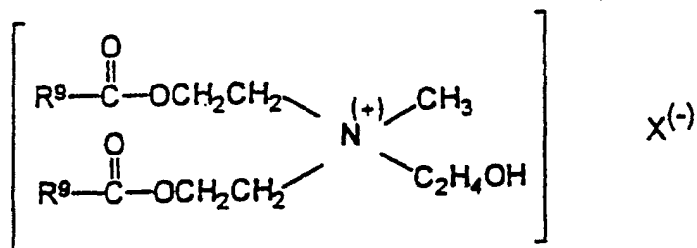
4. Composition d'adoucissement de tissus liquide aqueuse concentrée stable selon la revendication 1, comprenant :

(A) un matériau adoucissant de tissus d'ammonium quaternaire biodégradable de type diester ayant pour formule :



dans laquelle chaque groupe Y est -O(O)C ou -C(O)O, mais pas -OC(O)O, et dans laquelle chaque groupe R est choisi dans le groupe constitué de groupes alkyle ou hydroxyalkyle en C₁-C₆; chaque groupe R² est choisi dans le groupe constitué d'un groupe hydrocarbyle ou hydrocarbyle substitué en C₁₂-C₂₂ tel que le (IV) de l'acide gras apparenté de ce groupe R² soit inférieur à 10; et dans laquelle X⁽⁻⁾ est un anion compatible; et

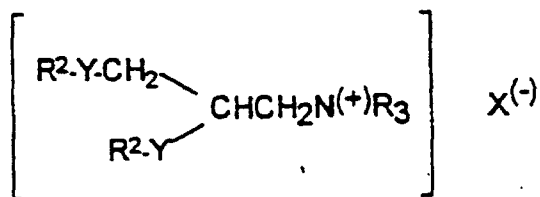
(B) un matériau adoucissant de tissus co-actif ayant pour formule :



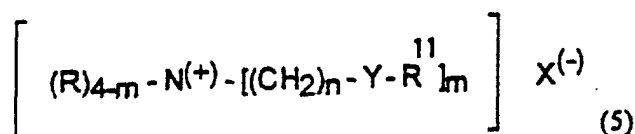
dans laquelle chaque groupe R⁹ est un groupe alkyle gras en C₁₅-C₁₇ tel que le (IV) de l'acide gras apparenté de ce groupe R⁹ soit de 20 à 100 et dans laquelle le groupe X⁽⁻⁾ a la même signification que ci-dessus.

5. Composition d'adoucissement de tissus liquide aqueuse concentrée stable selon la revendication 1, comprenant :

(A) un matériau adoucissant de tissus d'ammonium quaternaire biodégradable de type diester ayant pour formule :



dans laquelle chaque Y est —O(O)C ou —C(O)O-, mais pas -OC(O)O et dans laquelle chaque groupe R est choisi dans le groupe constitué de groupes alkyle ou hydroxyalkyle en C₁-C₆; chaque groupe R² est choisi dans le groupe constitué d'un groupe hydrocarbyle ou hydrocarbyle substitué en C₁₂-C₂₂ tel que le (IV) de l'acide gras apparenté de ce groupe R² soit inférieur à 10; et dans laquelle X⁽⁻⁾ est un anion compatible; et (B) un matériau adoucissant de tissus co-actif ayant pour formule :



dans laquelle chaque groupe R¹¹ est un substituant hydrocarbyle ou hydrocarbyle substitué à longue chaîne en C₁₂-C₂₂ tel que le (IV) de l'acide gras apparenté de ce groupe R¹¹ soit de 20 à 100; et chacun de m, n, Y, R et X⁽⁻⁾ a la même signification que ci-dessus, ou leurs mélanges.

6. Procédé de préparation d'une composition selon l'une quelconque des revendications 1 à 5, qui comprend les étapes consistant, en utilisant un pré-mélange fondu :

A. à pré-mélanger (A) un agent adoucissant de tissus d'ammonium quaternaire biodégradable de type diester et (B) un agent adoucissant de tissus secondaire ; et

B. à injecter le pré-mélange dans un bain d'acide et d'eau comprenant de préférence un acide choisi dans le groupe constitué des acides inorganiques minéraux et des acides organiques ayant pour formule R¹²COOH, dans laquelle R¹² est H ou un groupe alkyle en C₁-C₅, ou pour formule R¹³CH₂SO₃H, dans laquelle R¹³ est de l'hydrogène ou un groupe alkyle en C₁-C₄, et leurs mélanges; mieux encore dans lequel l'acide est l'acide phosphorique, l'acide chlorhydrique, l'acide citrique ou leurs mélanges ; de préférence dans lequel la concentration du bain d'acide et d'eau est jusqu'à 2 % ; et de préférence dans lequel le rapport molaire de l'acide à (B) est de 0:1 à 1,2:1 ; et

C. à effectuer un broyage à haut taux de cisaillement et à ajouter un électrolyte de préférence choisi dans le groupe constitué de sels de métaux des groupes IA et IIA du tableau périodique des éléments, de préférence le chlorure de calcium ou des sels d'ammonium.

7. Composition selon la revendication 8, dans laquelle ledit pré-mélange est combiné à un dispersant d'écume.