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US-A- 3 756 950

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Description

Technical Field

The present invention relates to concentrated fabric-softening compositions in liquid form, to be used in the rinse step of the laundry cycle.

Background

Fabric-softening compositions to be added in the rinse step of a laundry cycle are well known in the art and have found wide-scale commercial application.

In addition to achieving excellent fabric-softening performance, it has been, in recent years, the objective of fabric softener manufacturers to provide compositions which have an increased biodegradability.

Accordingly, ester-containing fabric-softening actives have been disclosed, to be used in replacement of, or in combination with, conventional quaternary softening actives. Such ester-containing actives include the imidazoline ester derivatives disclosed in e.g. GB 1,561,808 and further disclosed in e.g. EP-A-345 842.

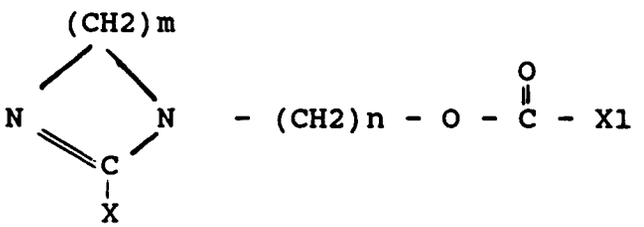
It has been found, however, that concentrated compositions containing said ester imidazoline compounds have a tendency to develop an odor off-note, on prolonged storage.

The present invention is based on the discovery that such a problem is surprisingly cured by the incorporation in said concentrated compositions of a small amount of chelating agent for heavy metal ions.

In U.S. Patent 3, 756,950 are disclosed fabric-softening compositions containing conventional quaternary ammonium softening actives and an acid salt of amino-carboxylic acid chelating agent, to decrease fabric yellowing.

Summary of the Invention

The present invention encompasses concentrated fabric-softening compositions containing at least 10% by weight, preferably 15 to 25% by weight of an imidazoline ester softening active of the formula :



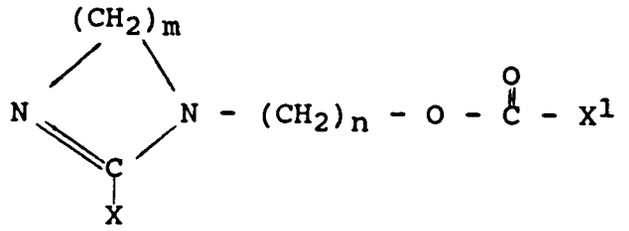
wherein X and X1 are, independently, C11-C21 hydrocarbyl groups, preferably C13-C17 alkyl, and m and n are, independently, from 2 to 4, preferably 2,

and a Bronstedt acid as levels such as to have a pH below 4, characterized in that, it contains a chelating agent for heavy metal ions, at levels of from 0.01% to 1%, preferably 0.05 to 0.2% of the composition.

Detailed Description of the Invention

Substituted imidazoline ester softening compound

The compositions of the present invention contain at least 10%, preferably from 15% to 25% of a substituted imidazoline ester softening compound having the formula

(b) 

wherein X and X^1 are, independently, C_{11}-C_{21} hydrocarbyl groups, preferably C_{13}-C_{17} alkyl, most preferably straight chain C_{17} alkyl, and m and n are, independently, from 2 to 4, preferably m and n are both 2. The total concentration of softener active cannot exceed about 40% by weight. It will be understood that substituents X and X^1 may optionally be substituted with various groups such as alkoxyl, hydroxyl, or can be branched, but such materials are not preferred herein. In addition X and/or X^1 may optionally be unsaturated (i.e., alkenyl groups). The preferred substituted imidazoline ester softening compounds will hereinafter be referred to as di-alkyl imidazoline ester compounds. Most preferred is ditallow imidazoline ester.

pH

The pH, measured in distilled water at 3% dilution of the present compositions should be not greater than 4, preferably from 2.5-4.

To achieve such a pH, a Bronstedt acid having a pKa value of 6 or less, is present in the present composition.

Examples of suitable acids include the inorganic mineral acids, carboxylic acids, in particular the low molecular weight (C_1-C_5) carboxylic acids, aromatic carboxylic acids, like benzoic acid, and alkylsulfonic acids.

Suitable ionorganic acids include HCl, HBr, H_2SO_4, HNO_3 and H_3PO_4. Suitable organic acids include benzoic acid, formic, acetic, methylsulfonic and ethylsulfonic acid. Preferred acids are phosphoric, formic, and methylsulfonic acid.

Liquid carrier

The compositions herein comprise a liquid carrier, e.g., water, preferably a mixture of water and a C_1-C_4 monohydric alcohol (e.g., ethanol, propanol, isopropanol, butanol, and mixtures thereof), isopropanol being preferred. These compositions comprise from 60% to 98%, preferably from 70% to 95% of the liquid carrier. Preferably, the amount of the C_1-C_4 monohydric alcohol in the liquid carrier is from 5% to 50% by weight of the softening compounds, the balance of the liquid carrier being water.

The chelating agent for heavy metal ions

The chelating agents are present at levels of from 0.01% to 1% of the compositions herein, preferably from 0.05 to 0.2%.

The chelating agents for heavy metal ions useful herein include any material known in the art for that purpose.

Suitable compounds include the following carboxylic acids and salts thereof : citric acid, tartaric acid, oxydisuccinic acid, ethylene diamine -N,N disuccinic acid, oxydiacetic acid, carboxymethyltartronic acid, carboxymethyloxysuccinic acid, tartrate monosuccinic acid/tartrate disuccinic acid blend at 80:20 weight ratio, as well as 2-dodecenyl succinic acid, and dodecyl succinic acid.

This category also includes polycarboxylic acids and salts thereof, such as polyacrylates and copolymers of maleic and acrylic acids.

Other suitable compounds are the following amino-carboxylic acids and salts thereof, as nitrilotriacetic acid, ethylene diamine tetracetic acid, cyclo ethylene diamine tetracetic acid, trans 1,2 diamino cyclohexane N,N,N',N' tetraacetic monohydrate (CHELCD), hydroxyethylenediamine tetracetic acid, diethylene triaminopentacetic acid, N-glyceryl imino diacetic acid, N(-2 hydroxypropyl) iminodiacetic acid;

Polyamino polyalkylene phosphonic acids and salts thereof such as ethylenediamine tetramethylenephosphonic acid and diethylene triamine pentamethylenephosphonic acid are also suitable as

chelating agents hereof.

Optional ingredients

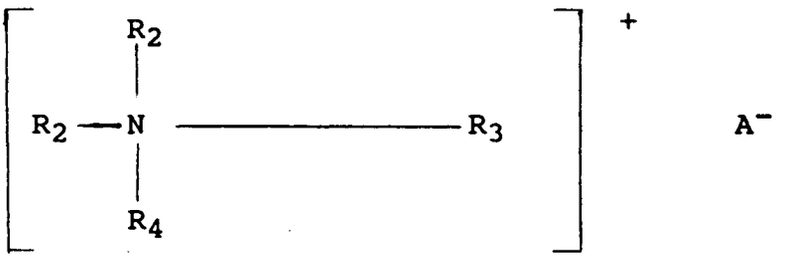
Fully-formulated fabric softening compositions may optionally contain, in addition to the substituted imidazoline ester softening compounds of the formula herein, the liquid carrier and the acid, one or more of the following ingredients.

Quaternary ammonium softening agents

The compositions of the present invention can further comprise a di(higher alkyl) quaternary ammonium softening agent. The compositions herein can contain from 0% to 25% (preferably from 0.1% to 10%) of the di(higher alkyl) quaternary ammonium softening agent.

The quaternary softening agents useful herein are selected from the group of

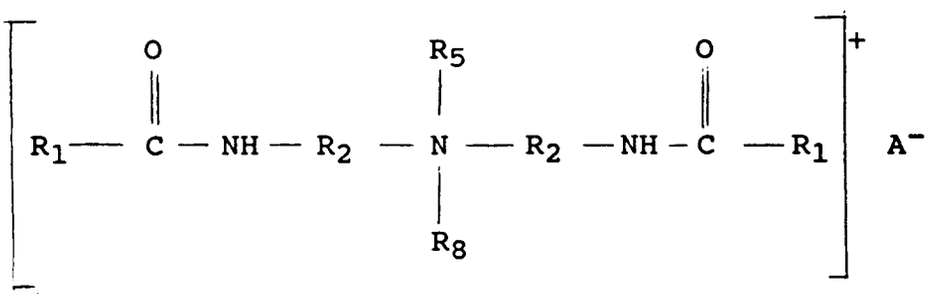
(i) acyclic quaternary ammonium salts having the formula:



wherein R_2 is an acyclic aliphatic C_{15}-C_{22} hydrocarbon group which may be interupted by ester groups. R_3 is a C_1-C_4 saturated alkyl or hydroxyalkyl group, R_4 is selected from R_2 and R_3, and A is an anion;

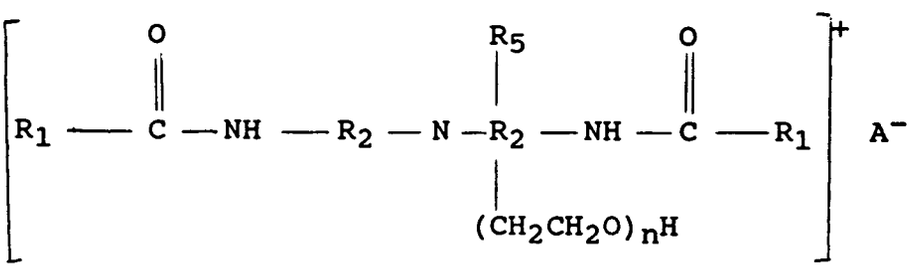
Rapidly biodegradable compounds of formula (i) where R_2, and possibly R_4, are interupted by ester groups, are disclosed in EPA 239 910.

(ii) diamido quaternary ammonium salts having the formula :



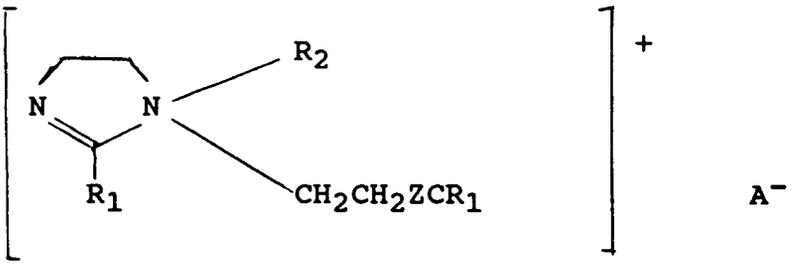
wherein R_1 is an acyclic aliphatic C_{15}-C_{22} hydrocarbon group, R_2 is a divalent alkylene group having 1 to 3 carbon atoms, R_5 and R_8 are C_1-C_4 saturated alkyl or hydroxyalkyl groups, and A^- is an anion;

(iii) diamido alkoxylated quaternary ammonium salts having the formula :



wherein n is equal to from 1 to 5, and R_1, R_2, R_5 and A^- are as defined above.

(iv) quaternary imidazolinium compounds having the formula :



wherein R_1 = C_{15}-C_{17} saturated alkyl, R_2 = C_1-C_4 saturated alkyl, Z = NH or O, and A^- is an anion.

Examples of Component (i) are the well-known dialkyldimethylammoniums salts such as ditallowdimethylammonium chloride, ditallowdimethylammonium methylsulfate, di(hydrogenated tallow) dimethylammonium chloride, dibehenyldimethylammonium chloride.

Examples of Component (ii) and (iii) are methylbis(tallowamidoethyl) (2-hydroxyethyl) ammonium methylsulfate and methylbis(hydrogenated tallowamidoethyl) (2-hydroxyethyl) ammonium methylsulfate, wherein R_1 is an acyclic aliphatic C_{15}-C_{17} hydrocarbon group, R_2 is an ethylene group, R_5 is a methyl group, R_8 is a hydroxyalkyl group and A is a methylsulfate anion; these materials are available from Sherex Chemical Company under the trade names Varisoft^R 222 and Varisoft^R 110, respectively.

Examples of component (iv) are 1-methyl-1-tallowamino-ethyl-2-tallowimidazolinium methylsulfate and 1-methyl-1-(hydrogenated tallowamidoethyl)methylsulfate.

The ratio of quaternary ammonium salt to cyclicamine should not exceed 10:1, and preferably does not exceed 2:1.

The aqueous dispersion of quaternary softening agent is prepared in a conventional way, typically by injecting the quaternary material in a molten form in a batch containing water, with continuous agitation.

Once this dispersion is prepared, the concentrated composition resulting from steps a) to d) will be added to it;

In case of a continuous process, said concentrated composition will, immediately after step d) be added to the aqueous dispersion of quaternary ammonium compound.

In an alternative and convenient way, the concentrated composition obtained after step d) will first be cooled to a temperature of from 15 to 25 °C, (room temperature) and then some time will be allowed for e.g. storage, shipment of the concentrated composition, before it is added to the aqueous dispersion of quaternary softening agent.

In any case, the viscosity of the finished product will be in a range of 20 to 500 cp, preferably 50 to 150 cp.

Optional co-softening agents

The co-softening agents for use herein can be selected from fatty acid esters of polyhydric alcohols having up to 8 carbon atoms such as described in DE-A-26 31 114. Examples of the like esters include sorbitan esters and glycerol esters such as sorbitan monostearate, sorbitan monooleate, glycerol mono-di- and tri-fatty acid esters wherein the acid is selected from stearic, oleic, lauric, capric, caprylic, caproic, valeric, butyric, propionic and acetic acid; an individual glycerol can be esterified by identical fatty acid groups or by mixed esters e.g. glycerol monostearatedioleate. Polyethyleneglycol esters monostearate, wherein the polyethyleneglycol moiety has a molecular weight in the range from 200 to 400 are also included in that class. Fatty acid esters of monohydric alcohols having at least 4 carbon atoms such as isobutyl stearate and ethyl hexyl stearate can also be useful.

Additional co-softening agents which can be used are : glycerol, diglycerol, ethylene glycol, propylene glycol, diethylene glycol, dipropylene glycol, dihexylene glycol, polyethylene glycol (MW 200-100.000), polypropylene glycol (MW 200-100.000), polyvinylalcohol, polyoxyethylenepolyoxypropylene copolymers, polypropylene glycol (MW 900), glucose methylether, butyldiglycolether, diethyleneglycolmonobutylether, propyleneglycolmonoethyl or ethyl ether, ethylene carbonate, propylene carbonate.

Useful also are alkylpolyglucosides of the general formula $R_2O(CnH_{2n}O)_t$ (glucosyl)$_x$ wherein R^2 is alkyl, alkylphenyl, hydroxyalkyl, hydroxyalkylphenyl and mixtures thereof wherein the alkyl chain has from 8 to 18

carbon atoms, t is from 0 to 2 and x from 2 to 7, can also be used in the composition. These glucosides exhibit desirable solvent properties and in addition can confer fiber benefits such as softness.

Lanolins and derivatives and paraffins having from 16 to 20 carbon atoms constitute another example of non-ionic agents which can be used if desired. Low melting oils from animal, vegetable or mineral origin are representative of this class of materials. Carnation oil^{R}, Jojoba oil^{R} and Sunflower oil are specific examples which are found to work.

Another important class of co-softening agents is represented by materials of the general formula :

R_1COOR_2

wherein R_1 is a straight or branched chain alkyl or alkenyl group having from 8 to 23 carbon atoms and R_2 is hydrogen or an alkyl or hydroxyalkyl group having 1-4 carbon atoms.

Highly preferred materials of this class are the C_{10} to C_{20} saturated fatty acids, especially lauric acid, myristic acid, palmitic acid and stearic acid.

Also suitable are linear fatty C_{8-18} akanol ethoxylates such as LUTENSOL® compounds available from BASF, as well as branched C_{8-18} akanols such as EXXAL compounds available from EXXON.

Clay materials such as the low ion-exchange-capacity ones describerd in EPA 150.531 can also be used.

The co-softening agents above can be used in combination with cation-active amines, namely primary, secondary and tertiary amines having, at least, one straight-chain organic group of from 12 to 22 carbon atoms. Preferred amines of this class are ethoxyamines, such as monotallow-dipolyethoxyamine, having a total of 2to 30 ethoxygroups per molecule. Suitable are also diamines such as tallow-N,N', N'-tris (2-hydroxyethyl)-1,3-propylenediamine, or C_{16-18}-alkyl-N-bis(2-hydroxyethyl) amines, examples of above amines are these sold under the trade name GENAMIN C, S, D, and T, by Hoechst.

The co-softening agents above are used at ratios of from 5:1 of 20:1 of quaternary softening agent to co-softening agent.

In the instance for fatty acids are used, a preferred ratio is from 8:1 to 10:1.

Further other optional ingredients for use herein include silicone components such as described in British Patent No. 1,549,180, emulsifiers, preservatives, germicides, dyes, bactericides, stabilizers, brighteners, opacifiers, photoactivators, copolymeric carboxylates having sequestering properties, soil release agents, bleaches, bleach activators, and anti-yellowing agents. Suitable polymeric soil-release agents are disclosed in EP-A-220 156.

These additives are normally incorporated at their conventional low levels e.g. from 0.001% to 5%, in the dilution step of the concentrates herein.

The following examples illustrate the present invention.

The following compositions were prepared :

Table 1

	Ex 1	**Ex 2**
Ditallow Imidazoline Ester	20	22
Perfume	0.80	0.80
Polydimethyl siloxane	0.2	0.2
Soil Release Polymer	0.2	0.2
Citric acid	0.1	0.05
CaCl2	0.2	0.2
water and minors	Balance	
pH 3%	3.0	

The compositions of Example 1 and 2 were stored for 3 weeks at 50 °C and compared with similar compositions not containing citric acid, stored under the same conditions; the compositions of Example 1 and 2 did not develop any perfume off-note, contrary to the reference compositions.

The following compositions are also prepared

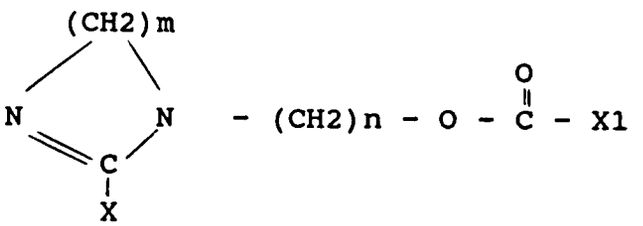
Table 2

	Ex 3	Ex 4	Ex 5	Ex 6
Dittalow Imidazoline Ester	21	22	23	23
Perfume	0.7	0.7	0.8	0.7
Polydimethyl siloxane	0.2	-	0.2	-
Soil Release Polymer	0.2	0.2	-	-
Dequest 2066*	0.1	-	-	-
EDTA disodiumsalt	-	0.1	-	-
CHELCD**	-	-	0.05	-
Tartaric Acid	-	-	-	0.1
water and minors	----------Balance ----------			
pH 3%	----------3.0 ----------			

* diethylene triamine pentamethylene phosphonic acid
** trans 1,2 diamino cyclohexane N,N,N',N' tetraacetic monohydrate

Claims

1. A concentrated fabric-softening composition containing at least 10% by weight of an imidazoline ester softening active of the formula :



wherein X and X1 are, independently, C11-C21 hydrocarbyl groups, preferably C13-C17 alkyl, and m and n are, independently, from 2 to 4, preferably 2,

and a Bronstedt acid as levels such as to have a pH below 4, characterized in that, it contains a chelating agent for heavy metal ions, at levels of from 0.01% to 1% of the composition.

2. The composition of claim 1 wherein said imidazoline ester is ditallow imidazoline ester.

3. The composition of claims 1-2 wherein said chelating agent is selected from the group of carboxylic acids, polycarboxylic acids, amino carboxylic acids, polyamino polyalkylenephosphonic acids, and salts thereof.

4. The composition of claim 3 wherein said chelating agent is selected from the group of citric acid, EDTA, and diethylene triamine pentamethylene phosphonic acid.

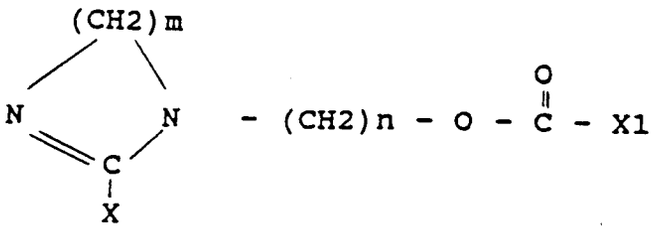
5. The composition of claims 1-4 wherein the level of chelating agent is from 0.05% to 0.2%.

6. The composition of claims 1-5 wherein the level of imidazoline ester is from 15% to 25%.

7. The composition of claims 1-6 wherein the pH of the composition is from 2.5 to 4.

Patentansprüche

1. Konzentrierte Textilweichmacherzusammensetzung, enthaltend mindestens 10 Gew.-% eines Imidazolinester-Weichmacherwirkstoffs der Formel:



worin X und X1 unabhängig voneinander C11-C21-Kohlenwasserstoffgruppen, vorzugsweise C13-C17-Alkyl, und m und n unabhängig voneinander 2 bis 4, vorzugsweise 2, sind,
und eine Brønstedt-Säure in solchen Anteilen, daß ein pH unterhalb 4 vorliegt, **dadurch gekennzeichnet**, daß sie einen Komplexbildner für Schwermetallionen in Anteilen von 0,01 bis 1 % der Zusammensetzung enthält.

2. Zusammensetzung nach Anspruch 1, wobei der Imidazolinester Ditalgimidazolinester ist.

3. Zusammensetzung nach den Ansprüchen 1 bis 2, wobei der Komplexbildner aus der Carbonsäuren, Polycarbonsäuren, Aminocarbonsäuren, Polyaminopolyalkylenphosphonsäuren und Salze davon umfassenden Gruppe gewählt ist.

4. Zusammensetzung nach Anspruch 3, wobei der Komplexbildner aus der Zitronensäure, EDTA und Diethylentriaminpentamethylenphosphonsäure umfassenden Gruppe gewählt ist.

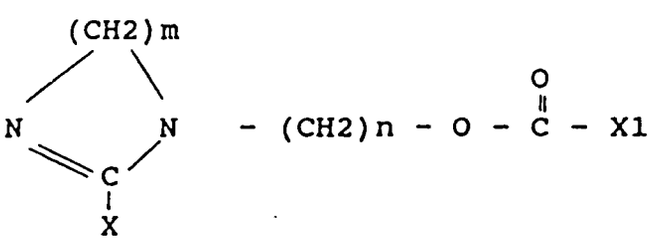
5. Zusammensetzung nach den Ansprüchen 1-4, wobei der Anteil des Komplexbildners 0,05 bis 0,2 % beträgt.

6. Zusammensetzung nach den Ansprüchen 1-5, wobei der Anteil des Imidazolinesters 15 bis 25 % beträgt.

7. Zusammensetzung nach den Ansprüchen 1-6, wobei der pH der Zusammensetzung 2,5 bis 4 beträgt.

Revendications

1. Composition d'assouplissant textile concentrée contenant au moins 10% en poids d'une substance active assouplissante ester d'imidazoline de formule:



dans laquelle X et X1 représentent indépendamment des groupes hydrocarbyles en C_{11}-C_{21}, de préférence des groupes alkyles en C_{13}-C_{17}, et m et n ont indépendamment une valeur de 2 à 4, de préférence une valeur de 2, et un acide de Bronstedt dans des proportions telles que l'on obtient un pH inférieur à 4, caractérisée en ce qu'elle contient un agent chélateur pour des ions de métaux lourds, dans des proportions de 0,01% à 1% de la composition.

2. Composition selon la revendication 1, dans laquelle ledit ester d'imidazoline est un ester de di-suif-imidazoline.

3. Composition selon les revendications 1-2, dans laquelle ledit agent chélateur est choisi dans le groupe constitué par les acides carboxyliques, les acides polycarboxyliques, les acides aminocarboxyliques, les acides polyaminopolyalkylènephosphoniques, et les sels de ceux-ci.

4. Composition selon la revendication 3, dans laquelle ledit agent chélateur est choisi dans le groupe constitué par l'acide citrique, l'EDTA, et l'acide diéthylènetriaminepentaméthylènephosphonique.

5. Composition selon les revendications 1-4, dans laquelle la proportion d'agent chélateur est de 0,05% à 0,2%.

6. Composition selon les revendications 1-5, dans laquelle la proportion d'ester d'imidazoline est de 15% à 25%.

7. Composition selon les revendications 1-6, dans laquelle le pH de la composition est de 2,5 à 4.