United States Patent Office 3,349,033 Patented Oct. 24, 1967

3,349,033

STABLE MICROBIOLOGICALLY ACTIVE LAUNDRY SOFTENER

Vincent Zuccarelli, Staten Island, N.Y., assignor, by mesne assignments, to Millmaster Onyx Corporation, New York, N.Y., a corporation of New York

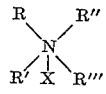
No Drawing. Filed Aug. 26, 1964, Ser. No. 392,307
5 Claims. (Cl. 252—8.75)

This invention relates to stable laundry softeners having microbiologically active properties.

An object of this invention is to provide a microbiologically active laundry softener for use in commercial or home laundering.

An object of the invention is to provide a stable laundry softener composition comprising a conventional organic fabric softener, a microbiologically active quaternary ammonium salt having a phenol coefficient of at least 200, and an emulsifying agent.

It is well known to the art to apply to laundered goods a quarternary ammonium salt as a cationic softener of the type:



wherein R and R′ represent alkyl radicals having 10 to 20 carbon atoms, R″ is a methyl radical, R‴ is a methyl, ethyl, an ethoxylated radical $(CH_2—CH_2O)_nH$ where n is an integer of at least one, and X is a chlorine, bromine, sulfate, methosulfate, or ethosulfate, by dissolving such a softener in the final rinse water. Similar materials have also been used by textile mills as softeners applied in the final rinse. In the preceding formula, R and R′ represent an alkyl radical or mixture of alkyl radicals having from 10 to 20 carbon atoms usually derived from tallow. Quaternary ammonium compounds of this type are sold in substantial volume and are used in the application set forth above, more particularly in the last rinse in household laundering operations. They impart to the treated goods a pleasant, soft hand and have found much favor with housewives.

The preferred alkyl radical, R, in these compounds is usually a mixture containing about 60 to 80% of stearyl and about 20 to 40% of cetyl radicals and is ordinarily derived from tallow, preferably by hydrogenation or by hydrogenation of its derivatives used as intermediates in the production of the quaternary ammonium compounds.

It has been found that mixtures of known fabric softeners of the type previously mentioned and known germicidal quaternary ammonium salts will not remain homogeneous for more than a few days or at most a few weeks even at ordinary temperatures. Such mixtures are quite unstable in hot weather, or when exposed to freezing. This is surprising, since it would have been expected that two quaternary ammonium salts, each of which contains at least one long carbon chain, would be compatible in a mixture because of their close structural relationship.

Di-(hydrogenated tallow) dimethyl ammonium methosulfate which is well known to the art makes an especially desirable fabric softener. However, in combination with a quaternary ammonium salt such as is effective as a fabric sanitizing agent, the stability of the mixture is poor; that is, on aging, heating or cooling, it separates into two phases.

I have found surprisingly that the addition of a small amount of an emulsifier, such as a long chain fatty alcohol, to the mixture of a quaternary ammonium germicide with a fabric softener, such as the di-(hydrogenated tallow) dimethyl ammonium methosulfate, causes the mixture to remain homogeneously emulsified over a

period of at least eight months, during which time it was stored at a temperature of 40° C. or 104° F. for a period of four months. It also causes the mixture to remain stable after several successive cycles of freezing and thawing followed by a period of storage at ordinary temperature for prolonged observation.

The fatty alcohols used in this invention suitable as the emulsifier may contain from 10 to 18 carbons in the chain. Topped coconut fatty alcohols, such as "Lorol 5" which is effective has an alkyl distribution of about 3% C_{10}, 61% C_{12}, 23% C_{14}, 11% C_{16} and 2% C_{18}. However, any aliphatic alcohol with a chain length of C_{10} to C_{18} may be used.

I may use in my composition any high-potency quaternary ammonium microbiologically active compounds having a phenol coefficient of at least 200 as determined by the standard method given in the "Official Methods of the Association of Agricultural Chemists," ninth edition, 1960. Typical examples of these quaternary ammonium compounds are alkyl dimethyl benzyl ammonium chloride in which the alkyl group may have from 8 to 22 carbon atoms, alkyl dimethyl substituted benzyl ammonium chlorides, in which the alkyl radical contains from 8 to 22 carbon atoms and in which the benzyl radical is substituted with one or more side chains containing from 1 to 4 carbon atoms such, for example, as methyl, dimethyl, trimethyl, tetramethyl, ethyl, diethyl, isopropyl and tertiary butyl or with one, two, or more, halogen atoms such as chlorine and bromine, alkyl dimethyl menaphthyl ammonium chloride and alkyl dimethyl tetrahydromenaphthyl ammonium chloride in which the alkyl radical contains from 8 to 22 carbon atoms, alkyl benzyl trimethyl ammonium chloride in which the alkyl radical contains from 8 to 22 carbon atoms and in which the aromatic nucleus of the benzyl radical may, if desired, be substituted by one or more methyl or other lower alkyl groups, alkyl phenoxy ethoxy ethyl dimethyl benzyl ammonium chloride in which the alkyl radical may be isooctyl or nonyl, and mixtures of the aforesaid quaternary ammonium compounds.

The following illustrative high-potency quaternary ammonium germicides are useful in my laundry softener compositions: dodecyl trimethyl ammonium chloride, cetyl trimethyl ammonium chloride, p-diisobutyl phenoxy ethoxy ethyl dimethyl benzyl ammonium chloride, dodecyl benzyl trimethyl ammonium chloride. Also any high-potency quaternary ammonium germicide of the alkyl dimethyl benzyl ammonium chloride compounds may be used, such as those used in the following examples sold by Onyx Chemical Corporation under the designation "BTC–2125" consisting of a mixture of benzalkonium salts of equal parts of alkyl dimethyl benzyl ammonium chloride (in which the alkyl distribution is 60% C_{14}, 30% C_{16}, 5% C_{12}, 5% C_{18}) and alkyl dimethyl ethylbenzyl ammonium chloride (in which the alkyl distribution is 50% C_{12}, 30% C_{14}, 17% C_{16}, 3% C_{18}). The following are the advantages of such mixture: (a) a high phenol coefficient; namely, 915 against *Staphylococcus aureus* and 612 against *Salmonella typhosa*; (b) exceptionally high hard water tolerance level of 850 parts per million; and (c) extensive testing under conditions simulating household laundering demonstrated that as little as 40 parts per million on the weight of the fabric was sufficient to prevent or arrest the growth of antibiotic-resistant strains of *Staphylococcus aureus*; and 100 parts per million on the weight of the fabric sufficed to prevent or control diaper rash caused by ammonia-producing organisms.

In a softener formulation such as is commonly purchased and employed by housewives, about 4.5% of active cationic softener is present. A few ounces of such a prep-

aration is customarily introduced into the final rinse water in a domestic washing machine, such rinse water varying in quantity from about 10 to about 20 gallons. The cationic softener is absorbed or absorbed on the fabric, depending on the chemical nature of the latter.

Accordingly, mixtures were prepared for comparative testing which contained 6% by weight of 75% active softener, and also 2% by weight of the quaternary ammonium germicide described above. To some of these preparations, 2% by weight of a long chain fatty alcohol was added.

These mixtures were aged at ordinary room temperature until evidence of phase separation appeared, or for as long as eight months. Other samples were aged similarly at a temperature of 40° C. to simulate hot weather conditions. Formulations which appeared reasonably stable were subjected to at least one cycle of freezing and thawing after which they were set aside for observation for a period of weeks for evidence of phase separation.

Three separate commercial brands of di-(hydrogenated tallow) dimethyl ammonium chloride type softeners were used as controls in these tests. Since they all performed almost exactly alike, they will be treated as a single example, and will be referred to as "D.T.C. Softener" for the sake of brevity. Similarly, di-(hydrogenated tallow) dimethyl ammonium methosulfate will be abbreviated to "D.T.Me. Softener."

The following examples, while not limiting the invention, will serve as illustrative embodiments:

EXAMPLE I

A household laundry softener concentrate of 75% active content of the di-(hydrogenated tallow) dimethyl ammonium methosulfate type was formulated as follows in parts by weight:

D.T.Me. Softener 75%	6.0
BTC–2125	2.0
Lorol 5	2.0
Water	90.0

The resulting emulsion has remained stable after eight months at ordinary temperatures (i.e. 20 to 25° C.). Other samples thereof were maintained at a temperature of 40° C. for at least four months. In no case was there any evidence of separation.

Other samples were subjected to freezing followed by thawing, several times repeated, after which they were set aside for some weeks for observation. At no time was there any separation.

Centrifuging for 15 minutes at high speed in an Adams "Safeguard Centrifuge" did not affect the stability of this emulsion.

EXAMPLE II

The formulation of Example I was altered by substituting for Lorol 5, a lauryl alcohol of commerce containing 80% of n-dodecanol. This formulation was appreciably less viscous in nature than its Lorol 5 counterpart. Under the above test conditions, it was fully as stable as that of Example I.

EXAMPLE III

Where 2% of long chain fatty alcohol was employed in Examples I and II, reducing the fatty alcohol to 1.5% by weight yielded stable emulsions. However, 1% by weight of fatty alcohol reduced the stability, and 3% of fatty alcohol while exerting a stabilizing effect increased the viscosity of the mixture to a point which could handicap marketing such a product.

EXAMPLE IV

Formulation as follows, omitting the fatty alcohol:

D.T.Me. Softener 75%	6.0
BTC–2125	2.0
Water	92.0

the emulsion broke down after two days at room temperature.

EXAMPLE V

Commercially available household laundry softeners containing 75% of di-(hydrogenated tallow) dimethyl ammonium chloride, and from three different manufacturing sources, were compounded separately according to the following formula:

D.T.C. Softener 75%	6.0
BTC–2125	2.0
Water	92.0

These mixtures were uniform emulsions when first prepared; phase separation was evident within 24 hours in every case.

EXAMPLE VI

The softener concentrates of Example V were formulated with fatty alcohol as follows:

D.T.C. Softener 75%	6.0
BTC–2125	2.0
Lorol 5	2.0
Water	90.0

These mixtures remained homogeneous at room temperature for two months, after which time some separation occurred.

Other samples of the same maintained at 40° C. broke down after one month.

EXAMPLE VII

The softener concentrates of Examples I and V were combined as follows:

D.T.Me. Softener 75%	4.0
D.T.C. Softener 75%	2.0
BTC–2125	2.0
Lorol 5	2.0
Water	90.0

These mixtures remained homogeneous for as long as six months at room temperature and four months at 40° C. Freezing and then thawing caused no damage.

The above examples demonstrate the stabilization of a germicidal di-hydrogenated tallow softener, either of the chloride but preferably of the methosulfate type by the addition of not less than 1% nor more than 3% of a fatty alcohol such, for example, as is contained in the mixture of such alcohols as are obtained by reducing coconut fatty acids by hydrogenation or otherwise, such softener containing as a component a quaternary ammonium germicide of the benzalkonium type, and specifically a modified benzalkonium germicide of high potency.

They also demonstrate that a fatty alcohol which is predominantly dodecanol is equally as effective as mixed coconut alcohols.

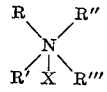
They also demonstrate that the presence of moderate amounts of chloride ion in the presence of a preponderance of methosulfate ion does not necessarily impair the stabilizing effect of fatty alcohol or alcohols.

The resistance of the above stabilized germicidal laundry softener to higher temperatures such as may be encountered in the summer season or to the cold of the winter season during shipment or shelf storage has also been demonstrated.

I claim:

1. A stable microbiocidally active laundry softener composition consisting essentially of a softeningly effective amount of (1) a quaternary ammonium fabric softener, (2) a microbiocidally effective amount of a quaternary ammonium salt having only one alkyl group of 8 to 22 carbon atoms attached to the quaternary nitrogen and having a phenol coefficient of at least 200, and (3) about 1 to 3% by weight of an emulsifying agent consist-

ing of a fatty alcohol having 10 to 18 carbon atoms in the chain, said fabric softener having the structure:



wherein R and R' are alkyl radicals having 10 to 20 carbon atoms, R'' is a methyl radical, R''' is a radical selected from the group of methyl, ethyl, and an ethoxylated radical having the structure $(CH_2—CH_2O)_nH$ where n is an integer of at least 1, and X is an anion selected from the group consisting of chlorine, bromine, sulfate and methosulfate.

2. A stable microbiologically active laundry softener composition as defined in claim 1 wherein the fabric softener is di-(hydrogenated tallow) dimethyl ammonium methosulfate.

3. A stable microbiologically active laundry softener composition as defined in claim 1 wherein the fabric softener is di-(hydrogenated tallow) dimethyl ammonium chloride.

4. A stable microbiologically active laundry softener

composition consisting essentially of a softeningly effective amount of di-(hydrogenated tallow) dimethyl ammonium salt, a microbiocidally effective amount of a benzalkonium salt having a phenol coefficient of at least 200, and about 1 to 3% by weight of a coconut fatty alcohol having 10 to 18 carbon atoms.

5. A stable microbiologically active laundry softener composition as defined in claim 4 wherein the benzalkonium salt is selected from the group consisting of alkyl dimethyl benzyl ammonium chloride and alkyl dimethyl ethyl-benzyl ammonium chloride, said alkyl having 8 to 22 carbon atoms.

References Cited

UNITED STATES PATENTS

3,093,591	6/1963	Freese	257—106
3,125,487	3/1964	Hutchinson	252—8.75
3,154,489	10/1964	DuBrow et al.	252—8.8 X
3,247,119	4/1966	Herrick et al.	252—106

LEON D. ROSDOL, *Primary Examiner.*

J. T. FEDIGAN, *Assistant Examiner.*