STABLIZED FABRIC SOFTENER COMPOSITION

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4 Claims. (Cl. 252—8.8)

This invention relates to a fabric softener and more particularly to a free-flowing liquid fabric softener which has an improved product stability and freeze recovery.

It has long been known in the art that softening agents may be applied in the laundering process to improve the softness or hand of clothes or other textile fabrics washed thereby. These softening agents are commonly used during the rinsing of the fabrics or are applied in a separate bath following the ordinary rinsing step.

In order to provide satisfactory softening effects to these fabric rinses it is essential that the softening agent be readily soluble or dispersible in the moderately warm or even cold water which may be used in the rinsing bath. Moreover, it is desirable that these agents be highly active and substantive to cloth when present in low concentrations in the rinsing bath. In addition, a highly active and readily dispersible product possesses the housewife with ease of application and enables her to achieve maximum softening effects with a minimum of product.

There are numerous liquid fabric softeners available today, many of which are based upon various long chain quaternary ammonium compounds as the active softening ingredient. Higher molecular weight quaternary amonium compounds have been found to be particularly useful for fabric softening because they are readily absorbed upon the fibers of cloth from dilute aqueous dispersions.

There are certain disadvantages, however, to the use of these liquid fabric softeners by the housewife and other persons engaged in the laundering process. Instability of the active ingredients of liquid softeners frequently results in separation of product upon standing. Moreover, certain additives such as coloring agents and perfume may also settle out during extended storage. These separation problems are particularly acute in the warmer climates where the softener product is exposed to relatively high temperatures.

Another disadvantage of the usual liquid softener is the tendency of the compositions to be very viscous unless the active softening ingredients are present only in low concentrations. A viscous product that is not readily pourable causes great inconvenience to the housewife. But a dilute composition that has satisfactory pourability generally requires large unwieldy product containers in order to provide the consumer with a reasonable number of active softening applications per container. The retail marketing of a dilute composition having a high proportion of water in a large product container results in the further disadvantage of high packaging and shipping costs.

The viscosity problem becomes particularly serious during the winter months when the liquid products are frequently exposed to low temperatures while in transit or in storage during the marketing process or while in the basement of the consumer. Many of the liquid products freeze or become extremely viscous at these low temperatures. Upon subsequent thawing or return to normal room temperature they show very poor recovery to their original state and remain viscous so that they are neither pourable nor readily dispersible in the rinsing bath.

Accordingly, it is an object of the present invention to provide a concentrated liquid fabric softener that is highly stable at the temperatures encountered during the marketing process and during laundering without loss of effective softening benefits.

It is another object to provide a fabric softener that does not separate on standing during long storage or shelf periods at moderately high temperatures and which can be readily poured directly into a rinsing bath without previous mixing and shaking or dilution by the consumer.

Another object is to provide a fabric softening composition that has a superior recovery when thawed after freezing so that it is thereafter pourable and readily dispersible in cold water.

Still a further object is to provide a highly active concentrated retail fabric softening composition which combines the benefits of ease of application with economical marketing costs.

Other objects and advantages of the present invention will be readily apparent from the detailed description that follows.

This invention is based on the discovery that small quantities of certain organic agents impart unique stabilizing properties to a fabric softening composition containing particular quaternary ammonium compounds. The stabilizing effects of this invention consist of a superior recovery of product flowability and dispersibility upon thawing after freezing, and non-separability of product throughout the temperature range of about 30°F. to 120°F.

The particular stabilizing agents used in this invention are the derivatives of diphenyl urea having the structure

\[
\begin{align*}
\text{Z:} & \quad \text{NH} - \text{C} - \text{NH} \\
\text{Y:} & \quad \text{Y1} - \text{Y2}
\end{align*}
\]

wherein one Z represents chlorine and the other Z represents a member selected from the group consisting of chlorine and the trifluoromethyl group and the Y atoms total one chlorine and two hydrogens.

Various derivatives of diphenyl urea (or carbamidine), including those used in the present invention, have previously been known to have excellent antisepctic or insecticidal properties. For example, U.S. Patent No. 2,346,398 discloses an antisepctic detergent composition containing various related polyhalogen substituted carbamidines having at least three halogens and U.S. Patent No. 2,745,874 discloses insecticidal derivatives of related diphenyl urea compounds having at least one trifluoromethyl group.

It has now been discovered that the use of small quantities of the diphenyl urea derivatives herein defined, ranging in amount from about 0.05% to about 0.9% by weight of the composition and preferably in an amount of 0.2% to 0.5% by weight, imparts an outstanding stability to aqueous mixtures of certain tetra alkyl ammonium salts and in particular the dialkyl dimethyl ammonium salts having the general structure

\[
\begin{align*}
\text{R:} & \quad \text{CH}_2 \\
\text{X:} & \quad \text{OH}_2
\end{align*}
\]

wherein R1 and R2 are long chain alkyl radicals having about 16-18 carbon atoms and X is the anion of a watersoluble salt such as chloride, bromide or ethsulfate. An example of a compound having the aforesaid general structure and preferred for its useful softening effects upon fabrics is di-tallow dimethyl ammonium chloride, "tallow" being the alkyl radicals corresponding to those in tallow fatty alcohol, i.e., a mixture of palmityl and stearyl.
3,216,944

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radicals. These quaternary ammonium compounds may be prepared by several well known means including the alkylation of alkyl secondary amines with alkyl halides such, for example, as described in U.S. Patent No. 2,775,617. Those tetra alkyl ammonium salts having two long chain C₆₋₈₃ alkyl groups have fabric softening characteristics superior to related compounds (e.g., those having shorter chain lengths or only one long chain alkyl group) but present greater stability problems than such related compounds.

The incorporation of the aforesaid diphenyl urea derivatives in aqueous softening products presented a difficult problem because they were known to be practically insoluble in water. Surprisingly, it was discovered that a particular method of formulating provided for the convenient incorporation of these diphenyl urea derivatives into aqueous fabric softener compositions. It was found that they can be added directly to the molten dialkyl dimethyl ammonium salts herein defined to produce a clear solution. This intermediate solution can then be mixed with agitation in a suitable amount of water, containing other additives desirable in softener compositions, in order to form the final fabric softener composition. In addition to the unexpected solubility of the diphenyl urea derivatives in the present softening agents, it was doubly surprising to find that the same compounds could not only provide antiseptic properties but also could produce the unique stabilizing effects herein disclosed.

The following examples are illustrative of the present invention but it will be understood that the invention is not limited thereto.

Example 1

A mixture of the following ingredients was prepared.

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Percent</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dialkyl dimethyl ammonium chloride—75% active ingredient dispersed in isopropanol and water, the dialkyl group being approximately 24% hexadecyl, 75% octadecyl and 1% octa-deceny1</td>
<td>7.0</td>
<td></td>
</tr>
<tr>
<td>3,4,4'-trichlorocarbanilide</td>
<td>7.0</td>
<td></td>
</tr>
<tr>
<td>The condensation product of 9 mol of ethylene oxide with 1 mol of nonyl phenol</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>Color</td>
<td>0.003</td>
<td></td>
</tr>
<tr>
<td>Perfume</td>
<td>0.25</td>
<td></td>
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<tr>
<td>Water</td>
<td>91.247</td>
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</table>


The "Arquard 2HT-75" was first heated until it melted at about 120° F. The trichlorocarbanilide was then dissolved in the molten "Arquard 2HT-75" to form mixture A which was a clear solution. The color, perfume and nonyl phenol ethylene oxide condensate were then added to the water to form mixture B. While still warm, mixture A was added to mixture B with agitation to form a final composition having a viscosity of 100 cps. at room temperature. The minor additives in mixture B, though having no material effect upon the softening power and stability of the final mixture, were used for certain desirable effects. For example, the Polar Brilliant Blue produced a whitening effect on laundered clothing, the perfume added a pleasant odor to the softener and the nonyl phenol ethylene oxide condensate reduced the viscosity of the final mixture. The separation was noticeable when the final mixture was subjected to a storage temperature of 100° F. for a period of one year, whereas a similar product containing no trichlorocarbanilide produced substantial separation of ingredients in the form of a conspicuous layer at the bottom of the product container after a storage period of only one month at 100° F. The stabilized final mixture froze when subjected to a temperature of 20° F. for a storage period of 24 hours. But upon thawing to room temperature its flowability returned to normal and it was readily pourable, whereas a similar product containing no trichlorocarbanilide had a thick gel-like consistency and could not be poured when allowed to thaw to room temperature after similar freezing. 4 grams of the stabilized mixture were readily dispersed in one gallon of cold water and laundered clothing articles rinsed therein were found to be soft to the touch and had a desirable fluffed-up appearance.

Example 2

One part by weight of 3,4,4'-trichlorocarbanilide was added to 14 parts by weight of molten "Arquard 2HT-75" (ditallow dimethyl ammonium chloride). This mixture was then added to 185 parts by weight of water with agitation to form a stable and pourable suspension having a viscosity (Brookfield) of about 350 cps. at room temperature. Four test samples of this mixture showed no signs of product separation when kept at temperatures of 80°, 100°, 120° and 140° F., respectively, during storage periods of five months, whereas a similar product containing no trichlorocarbanilide produced substantial separation of ingredients in the form of a conspicuous layer at the bottom of the product container after a storage period of only one week at 100° F. When used at a concentration of 4 grams per gallon of water for rinsing laundered clothes, the stabilized mixture imparted a desirable soft feel to the clothes. The trichlorocarbanilide also provided an effective antiseptic action on the clothes.

Distearil dimethyl ammonium bromide can be substituted for the "Arquard 2HT-75" in Example 2 with substantially equal results.

Example 3

Three parts by weight of 4,4'-dichloro-3-(trifluoro-methyl) carbanilide were added to 70 parts by weight of molten "Arquard 2HT-75" (ditallow dimethyl ammonium chloride). This mixture was then added to 927 parts by weight of water with agitation. The final mixture was stable and pourable. Four test samples of this mixture showed no signs of product separation when kept at temperatures of 80°, 100°, 120° and 140° F., respectively, during storage periods of four months. When used at a concentration of 4 grams per gallon of water for rinsing laundered clothes, the stabilized mixture imparted a soft feel to the clothes.

Dipalmityl dimethyl ammonium ethosulfate can be substituted for the "Arquard 2HT-75" in Example 3 with substantially equal results.

In the above examples various amounts of the active softening agent herein defined can be used ranging from about 3% to about 8% by weight of the total composition. Amounts less than about 3% are relatively dilute and do not satisfy the objective of producing an effective concentrated fabric softener composition. Amounts greater than 8% tend to become too viscous for satisfactory usage even with the use of diphenyl urea derivatives. It is preferable to use about 4-6%. The active softening agents can also be incorporated into the softening composition herein defined by adding them in their pure form instead of in the form of aqueous dispersions, such as the aforesaid "Arquard 2HT-75," with substantially equal results.

The amount of diphenyl urea derivative used can vary from about 0.05% to about 0.9% by weight of the total composition. The amount used will depend somewhat upon the amount of active softening agent used and the expected storage conditions. Levels greater than about 0.9% tend to increase the viscosity of the final softener composition without noticeably improving its stability, whereas levels less than about 0.05% produce no noticeable stabilizing effects. It is preferable to use about 0.2-0.5%.
Although, as previously described, the use of the diphenyl urea derivatives herein defined achieves the marked improvement in stability of product and freeze recovery, it is advantageous to incorporate in the softening composition a small amount of a water-soluble nonionic surfactant for its fluidizing effects. The use of a nonyl phenol condensed with 9 molecules of ethylene oxide has been found to be satisfactory for reducing the viscosity of the softener composition of this invention although a sufficiently pourable product can be obtained without its incorporation.

Various minor ingredients, such as color, perfume, preservative and other antiseptic agents may be added in the usual small amounts without adversely affecting the basic and novel characteristics of the composition. For example, it has been found beneficial in achieving fabric whiteness to add a small amount from about 0.001% to about 0.04% of the bluing agent “Polar Brilliant Blue GAW 180%,” reported to have the formula \( \text{HN} \cdot \text{H} \cdot \text{N} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{S} \cdot \text{Na} \), for its bluing effects on the fabrics rinsed.

The stabilized fabric softener compositions of this invention can be prepared by dissolving about 1 to about 18 parts by weight of the diphenyl urea derivatives herein defined in about 60 to about 160 parts by weight of the molten dialkyl dimethyl ammonium salts herein defined to form a clear intermediate solution and dispersing about 3 to about 9 parts by weight of the said intermediate solution in about 91 to about 97 parts by weight of an aqueous system which can contain the aforesaid minor ingredients. A very satisfactory composition can be prepared according to this procedure by using about 10 parts by weight of 3,4,4'-trichlorocarbanilide with about 105 parts by weight of ditallow dimethyl ammonium chloride and by using about 5.75 parts by weight of the intermediate solution prepared thereby with about 94.25 parts by weight of the aqueous system.

What is claimed is:

1. The process of making a stable fabric softener composition which comprises dissolving about 1 to about 18 parts by weight of a derivative of diphenyl urea selected from the group having the general formula

\[
\begin{align*}
\text{Z}_1 \quad \text{Z}_2 \\
\text{Z}_2 \quad \text{Z}_1
\end{align*}
\]

wherein one \( Z \) represents chlorine and the other \( Z \) represents a member selected from the group consisting of chlorine and the trifluoromethyl group and the \( Y \) atoms total one chlorine and two hydrogens in about 60 to about 160 parts by weight of a molten quaternary ammonium compound selected from the group having the general formula

\[
\begin{align*}
\text{R}_1 \quad \text{N} \cdot \text{CH}_2 \cdot \text{N} \cdot \text{O} \cdot \text{Y} \\
\text{R}_1 \quad \text{N} \cdot \text{CH}_2 \cdot \text{N} \cdot \text{O} \cdot \text{Y}
\end{align*}
\]

wherein \( R_1 \) and \( R_2 \) are alkyl groups having from about 16 to about 18 carbon atoms and \( X \) is the anion of a water-soluble salt and is selected from the group consisting of chloride, bromide and ethosulfate to form a clear intermediate solution and mixing about 3 to about 9 parts by weight of the said intermediate solution in about 91 to about 97 parts by weight of water to form the final stabilized fabric softener composition.

2. The process of making a stable fabric softener composition which comprises dissolving about 10 parts by weight of 3,4,4'-trichlorocarbanilide in about 105 parts by weight of ditalloy dimethyl ammonium chloride to form a clear intermediate solution and mixing about 5.75 parts by weight of the said intermediate solution in about 94.25 parts by weight of water to form the final stabilized fabric softener composition.

3. The process of claim 1 wherein the quaternary ammonium compound is ditalloy dimethyl ammonium chloride and the derivative of diphenyl urea is 3,4,4'-trichlorocarbanilide.

4. The process of claim 1, wherein the quaternary ammonium compound is ditalloy dimethyl ammonium chloride and the derivative of diphenyl urea is 4,4'-dichloro-3-(trifluoromethyl)carbanilide.

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<th>Class</th>
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JULIUS GREENWALD, Primary Examiner.