LIQUID LAUNDRY DETERGENTS AND A PROCESS FOR PREPARING SAME

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No Drawing. Filed Mar. 15, 1971, Ser. No. 124,601

Int. Cl. C11D 1/18

U.S. Cl. 252—543

5 Claims

ABSTRACT OF THE DISCLOSURE

A liquid detergent composition comprising a solubilized stiblene brightener system, a surfactant, the sodium salt of nitroisotriaetic acid, and a hydrotrope. Moreover, a process of preparing said brightener system comprising solubilizing the stiblene brighteners with a water miscible solvent and heat is also described.

The subject invention is directed to clear liquid detergent compositions containing chemical bleaching and/or brightening agents, in specific, are well known in the art. Such compositions have in the past comprised a surfactant material in combination with a brightening or bleaching agent which was dispersed therein. It is noted, however, that such dispersion resulted in numerous desirable characteristics such as uniform distribution and, further, with uniform concentration of the optical brightener contained in such compositions.

Moreover, chemical bleaching or brightening agents or a combination of chemical bleaching agents or a combination of brightening agents, and surfactants for washing or rinsing or washing and rinsing or rinsing and subsequent dilution with large quantities of rinse or wash water so as to result in an effective concentration of brightener.

Further, when applied to the fabric, the fluorescent compound must be evenly and uniformly distributed since, otherwise, the appearance of the treated fabric is spotty and non-uniform.

In the past it has been found that in order to obtain the desired uniform, level distribution, of the optical brightener such brightener must be dispersed in a suitable solution. Where the brightener is a water insoluble compound, such requirement poses the additional problem of solubilization of such brighteners. In general, for practical reasons of economy and because of the ultimate mode of solution, brightening compositions have, in the past, been packaged and sold in a highly concentrated form. Therefore, in the past solubilization of water insoluble fluorescent compounds has been extremely difficult problem especially in light of the fact that high concentrations of such compounds had to be incorporated into a concentrate for subsequent dilution with large quantities of rinse or wash water so as to result in an effective concentration of brightener.

Therefore, it is an object of the instant invention to provide a clear liquid detergent composition. Another object of the instant invention is to provide a clear liquid detergent composition containing an effective amount of optical brighteners. Yet another object of the instant invention is to provide a process for incorporating optical brighteners into a liquid detergent system.

A still further object of the instant invention is to provide a process for incorporating optical brighteners into a detergent system which includes an anionic surfactant, which process comprises solubilizing optical brighteners with a solvent therefor. These and other objects of the instant invention will become more evident from the following more detailed description thereof.

As noted, the subject invention is directed to a clear liquid detergent which includes therein optical brighteners. Such optical brighteners are generally classified as anionic fluorescent dyes which serve to impart increased fluorescence and/or optical brightness to both hydrophilic and hydrophobic fibers. Typical representative examples thereof include:

1. disodium N,N'-bis-(2-dithanolamino-4-phenylamino-1:3:5-triazyl)-(6)-(4'-diamino stibene 2:2'-disulfonate,
2. disodium N,N'-bis-(2-morpholino-4-phenylamino-1:3:5-triazyl)-(6)-(4'-diamino stibene 2:2'-disulfonate,
3. disodium N,N'-bis-(2,4-diphenylamino-1:3:5-triazyl)-(6)-(4'-diamino stibene 2:2'-disulfonate,
4. disodium 4,4'-bis-(6-N-phenyl-N-hydroxyethylamino)-s-triazin-2-ylamino) stibene disulfonate, disodium 4,4'-bis-(4-phenyl-2,1,3-triazole-2-yl) stibene disulfonate,
4. disodium 4,4'-bis-[4-anilino-6-(N-methyl-N-hydroxyethy lamino)-s-triazin-2-ylamino) stibene disulfonate, and
5. sodium 2-(stilbene-4')-naphtho-1',2',4',5',1,2,3-triazole-2'-sulfonate.

Other suitable anionic fluorescent dyes are disclosed in South African Pat. No. 66/2892 and in U.S. Pat. Nos. 2,376,743, 2,612,501, 2,762,801, 2,784,183, and 3,012,971, the disclosures of which are incorporated herein as references. Also suitable for use in connection with the novel clear liquid detergent composition of the instant invention are other well known stibene type brighteners in addition to those noted above.

In addition to the brightening agent, the subject clear liquid laundry detergents also include anionic and/or nonionic type surfactants. Suitable anionic detergents include materials such as alkylbenzenesulfonic acid and its saturated, and compounds of the formula alkyl-phenyl-SO-3M, wherein alkyl is an alkyl radical of a fatty acid and M is hydrogen or an alkali metal, which compounds comprise a well-known class of anionic detergents and include sodium dodecylbenzenesulfonate, sodium dodecylbenzenesulfonate, sodium laurylbenzenesulfonate, sodium cetylbenzenesulfonate. Others are the alkali metal salts of the higher alkylsulfonic acids and the alkali metal dialkyl sulfoisocyanates, e.g., sodium dioctylsulfoisocyanate; and sodium dioctylsulfoisocyanate; sodium dioctylsulfoisocyanate; sodium oleoyl-p-anisidesulfonate; sodium tetradesacanesulfonate; sodium diisopropylphenylsulfonate; sodium octylphenoxyethoxyethanesulfate, etc.; and the alkali metal alkyl sulfates, e.g., sodium lauryl sulfate and the like.

Among the above-noted alkylbenzenesulfonic acid and salts thereof, the preferred compounds included those...
which are biodegradable and which are particularly characterized by a linear alkyl substituent of from C₁₅ to C₂₂ and preferably from C₁₅ to C₂₀. It is, of course, understood that the carbon chain length represents, in general, an average chain length since the method for producing such products usually employs alkylating reagents of mixed chain length. It is clear, however, that substantially pure olefins as well as alkylating compounds used in other techniques can and do give alkylated benzene sulfonates wherein the alkyl moiety is substantially (i.e. at least 99%) of one chain length, i.e., C₁₅, C₁₆, C₁₇, or C₁₈. The linear alkyl benzene sulfonates are further characterized by the position of the benzene ring in the linear alkyl chain, with any of the position isomers (i.e. alpha to omega) being operable and contemplated.

The linear alkyl benzene sulfonates are generally and conveniently prepared by sulfonating the corresponding alkyl benzene hydrocarbons which in turn may be prepared by alkylating benzene with a linear alkyl halide, a 1-alkene or a linear primary or secondary alcohol. Pure isomers (of the 1-phenyl isomer) are prepared by reduction of the acylated benzene (alkyl phenyl ketone) using a modification of the Wolff-Kishner reaction. The 2-phenyl isomer is obtained from n-undecyl phenyl ketone and methyl magnesium bromide to form the tertiary alcohol which is dehydrated to the alkene and then hydrolyzed. The 5-phenyl isomer is obtained similarly from a n-heptyl phenyl ketone and n-butyl magnesium bromide. The other isomers are obtained in a similar manner from the appropriate n-alkyl phenyl ketone and n-alkyl magnesium bromide.

In addition to the benzene sulfonates one may also employ the lower alkyl (C₁ to C₄) analogs of benzene such as toluene, xylene, the trimethyl benzenes, ethyl benzene, isopropyl benzene and the like. The sulfonates are generally employed in the water soluble salt form which include as the cation, the alkali metals, ammonium, and lower amine and alkalanolamine.

Examples of suitable linear alkyl benzene sulfonates:
- sodium n-decyl benzene sulfonate
- sodium n-dodecyl benzene sulfonate
- sodium n-tetradecyl benzene sulfonate
- sodium n-pentadecyl benzene sulfonate
- sodium n-hexadecyl benzene sulfonate
and the corresponding lower alkyl substituted homologues of benzene as well as the salts of the cations previously referred to. Mixtures of these sulfonates may, of course, also be used with mixtures which may include compounds wherein the linear alkyl chain is smaller or larger than indicated herein provided that the average chain length in the mixture conforms to the specific requirements of C₁₀ to C₂₀.

The linear paraffin sulfonates are also a well-known group of compounds and include water soluble salts (alkali metal, amine, alkalanolamine, and ammonium) of:
- 1-decane sulfonic acid
- 1-dodecane sulfonic acid
- 1-tridecane sulfonic acid
- 1-tetradecane sulfonic acid
- 1-pentadecane sulfonic acid
- 1-hexadecane sulfonic acid
as well as the other position isomers of the sulfonic acid group.

In addition to the paraffin sulfonates illustrated above, others with the general range of C₁₀ to C₂₀ alkyis may be used, with the most preferable range being from C₁₂ to C₂₀.

The linear alkyl sulfates which are contemplated in this invention comprise the range of C₁₀ to C₂₀. Specific examples include sodium decyl sulfate; sodium n-dodecyl sulfate; sodium n-hexadecyl sulfate; sodium n-octadecyl sulfate; and the ethoxylated (1 to 100 moles ethylene oxide) derivatives; and, of course, the other water-soluble salt-forming cations mentioned above.

Also useful in conjunction with the instant invention are nonionic detergents such as alkaryl polyglycol ether sulfates; sodium n-octyl ether sulfate, sodium n-decyl ether sulfate; sodium n-dodecyl ether sulfate; sodium n-tetradecyl ether sulfate; sodium n-pentadecyl ether sulfate; sodium n-hexadecyl ether sulfate; sodium n-octadecyl ether sulfate; and the ethoxylated (1 to 100 moles ethylene oxide) derivatives; and, of course, the other water-soluble salt-forming cations mentioned above.

The composition of the instant invention may also include, in addition to the fabric softening compounds and conventional anionic, cationic, and amphoteric detergent compositions, builders, brighteners, hydroxropes, germicides, soil suspending agents, anti-redeposition agents, antioxidants, bleaches, coloring materials (dyes and pigments), perfumes, water-soluble alcohols, foam boosters, non-detergent alkali metal benzene sulfonates, etc.

Moreover, the subject composition may, in addition, include a builder material. Preferably, the builder material is a water-soluble salt of an amino carboxylic acid such as, for example, nitrilo triacetic acid. Preferably, one employs the trisodium salt thereof as the sole builder in connection with the instant composition.

In addition to the above ingredients one may as previously delineated employ hydroxropes in connection with the compositions of the instant invention. The useful hydroxropes include such compounds as, sodium xylene sulfonate, potassium xylene sulfonate, sodium and potassium toluene sulfonates, in the position isomers thereof, and ethyl benzene sulfonate.

As previously noted, the subject invention is also directed to a process for incorporating the above-noted optical brighteners into a liquid detergent composition comprising the other materials discussed above so as to provide a clear liquid laundry detergent having brightening characteristics. According to the novel process of the instant invention, the brightening materials are first incorporated into a solvent therefor, such as an ethylene glycol ether or dimethyl sulfoxide. Representative ethylene glycol ethers include ethylene glycol monomethyl ether, ethylene glycol monoethanol ether, ethylene glycol monopropyl ether, ethylene glycol monoethyl ether, diethylene glycol monobutyl ether, diethylene glycol monoisopropyl ether, diethylene glycol monoethyl ether, and triethylene glycol diethyl ether.

Representative of ethoxylated fatty alcohols which may be utilized in connection with the instant invention, include in addition to those noted above the following fatty alcohols containing from about 2 to about 200 moles of ethylene oxide, such as, for example, octanol, nonanol, decanol, undecanol, dodecanol, tridecanol, tetradecanol, pentadecanol, and the like. Subsequent to incorporating the optical brighteners into the above noted solvent system comprising an ethylene glycol ether and/or dimethyl sulfoxide and an ethoxylated fatty alcohol with heat, the said brightener system may then be incorporated into the remaining ingredients so as to provide a clear liquid laundry detergent formulation.

Preferably, one employs from about 0.01% to about 0.5% brightener based on the weight of the total active
ingredients in the final detergent formulation. While the proportions of the remaining ingredients are not critical to the instant invention, it is noted that generally one may employ from about 1 to about 40% total surfactant ingredient, from 1 to about 50% builder, from about 1 to about 40% hydrotropc, from about 1 to about 20% ethoxylated fatty alcohol, with the remaining proportions being made up of additive materials such as perfumes, dyes, and the like, and water.

The instant invention will now be illustrated by the following more detailed examples thereof. It is to be noted, however, that the instant invention is not deemed as being limited thereto.

**EXAMPLE 1**
A clear liquid detergent composition was prepared comprising:

<table>
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<tr>
<th>Ingredient</th>
<th>Wt. percent</th>
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<tr>
<td>Sodium tridecyl benzene sulfonate</td>
<td>2.5</td>
</tr>
<tr>
<td>Ethoxylated fatty alcohol</td>
<td>7.5</td>
</tr>
<tr>
<td>Nitrotriacetic acid trisodium salt</td>
<td>20.0</td>
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<tr>
<td>Ethylene glycol monobutyl ether</td>
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<td>4,4'-bis - (2,4-diamino-s-triazinyl-(6)-amino)-stilbene-2,2'-disulfonic acid, disodium salt</td>
<td>0.20</td>
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<tr>
<td>4,4'-bis - (2-(3-methylpyrrolidino)-4-anilino-s-triazinyl-(6)-amino)-stilbene-2,2'-disulfonic acid, disodium salt</td>
<td>0.07</td>
</tr>
<tr>
<td>Ammonium cumene sulfonate</td>
<td>11.80</td>
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</table>

WATER AND OTHER QUANTITY SUFFICIENT TO MAKE UP 100%.

The above composition was prepared by first mixing the stilbene brighteners with the ethoxylated fatty alcohol and the ethylene glycol monobutyl ether. The resulting mixture was then heated to 120 °F. for fifteen minutes and subsequently added to a mixture of deionized water, nitrotriacetic acid trisodium salt, and ammonium cumene sulfonate. The remaining ingredients were then added and the product which resulted was found to be a clear liquid detergent composition.

**EXAMPLE 2**
The composition of Example 1 was again prepared, substituting sodium xylene sulfonate for the ammonium cumene sulfonate therein. The composition was prepared as in Example 1 and found to produce a stable clear liquid detergent composition containing brightener materials.

**EXAMPLE 3**
The composition of Example 1 was again prepared, substituting 2% by weight dimethylsulfoxide for the ethylene glycol monobutyl ether employed therein. Once again, the composition which resulted was found to be a clear liquid.

**EXAMPLE 4**
The composition of Example 1 was again prepared substituting for the brighteners utilized therein mixtures of a disodium N,N'-bis-(2-dihexanolamino-4-phenylamino-1:3:5-triazyl-[6(1)])-4,4'-diamino stilbene 2:2'-disulfonate, disodium N,N'-bis -(2,4-diphenylamino-1:3:5-triazyl-[6(1)]-4:4'-diamino stilbene 2:2'-disulfonate, disodium 4,4'-bis-[4-anilino-6-(N-methyl-N-hydroxyethylamino)-s-triazin-2-y lamino] stilbene disulfonate, disodium 4,4'-bis-(phenyl-2, 1,3-triazole-2yl) stilbene disulfonate, di sodium 4,4'-bis-(4-anilino-6-(N,N'-methyl-2-hydroxethylamino)-s-triazin-2-y lamino) stilbene disulfonate and sodium 2 - (stilbyl - 4') - naphtho - 1,2',4:4', 1,2,3-triazole-2'-sulfonate. As a result of the foregoing substitutions, clear liquid detergents were produced.

As is readily apparent from the foregoing, the subject invention provides a process whereby stilbene type brighteners are readily solubilized and incorporated into a clear liquid detergent composition for use in the washing and brightening of fabrics.

What is claimed is:

1. A clear liquid brightener detergent composition consisting essentially of from about 1% to about 40% of a water-soluble anionic organic detergent, from about 1% to about 50% of a water-soluble salt of an amino carboxylic acid, from about 1% to about 40% of a hydrotrope selected from the group consisting of the sodium, potassium and ammonium salts of xylene sulfonic acid, toluenesulfonic acid, ethyl benzene sulfonic acid and cumene sulfonic acid from about 1% to about 20% of an ethoxylated fatty alcohol having from about 8 to about 15 carbon atoms, from about 0.01% to about 5% of an anionic stilbene optical brightener and from about 1% to about 20% of a compound selected from the group consisting of ethylene glycol ethers and dimethyl sulfosuccinate, said ethylene glycol ethers selected from the group consisting of ethylene glycol monoalkyl ethers, ethylene glycol dialkyl ethers and ethylene glycol trialkyl ethers, said ethers having an alkyl group of from about 1 to about 4 carbon atoms, and water.

2. The composition of claim 1 wherein the hydrotropic is ammonium cumene sulfonate.

3. The composition of claim 1 wherein the water-soluble salt of an amino carboxylic acid is the trisodium salt of nitrotriacetic acid.

4. The composition of claim 1 wherein the ethylene glycol ether is ethylene glycol monobutyl ether.

5. A process for preparing a clear liquid brightener detergent composition which comprises mixing from about 0.01% to about 0.5% of an anionic stilbene brightener with from about 1% to about 20% of a compound selected from the group consisting of ethylene glycol ethers and dimethyl sulfosuccinate, said ethylene glycol ethers selected from the group consisting of ethylene glycol monoalkyl ethers, ethylene glycol dialkyl ethers and ethylene glycol trialkyl ethers, said ethers having an alkyl group of from about 1 to 4 carbon atoms and from about 1% to about 20% of an ethoxylated fatty alcohol having from about 8 to 15 carbon atoms, heating the mixture to a temperature of from about 75° to about 160° F. for a period of time sufficient to solubilize the brightener in the liquid composition and subsequently mixing the solubilized brighteners with from about 1% to about 40% of an anionic water-soluble detergent and water.

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WILLIAM E. SCHULZ, Primary Examiner

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