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3,485,761

PRODUCTION OF DETERGENT COMPOSITIONS CONTAINING FINELY DISPERSED OPTICAL BRIGHTENERS

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2 Claims

ABSTRACT OF THE DISCLOSURE

A process for producing an anionic detergent composition with brightening properties by spraying a mixture of a relatively water-insoluble brightener dispersed in fatty acids onto a granular anionic detergent mixture.

This is a continuation of a pending application Ser. No. 375,326, filed June 15, 1964, now abandoned.

This invention relates to new compositions of matter comprising specific detergent brighteners which are relatively water-insoluble and to processes for preparing detergent compositions utilizing said new compositions of matter.

Certain optical brighteners which are relatively insoluble in water are particularly effective for use in detergent compositions which are used to wash, e.g., such synthetic textiles as "nylon" and "Dacron." It has been discovered that detergent compositions comprising these relatively water-insoluble optical brighteners must contain the optical brightener in a highly dispersed form if maximum deposition of the optical brightener on the article to be cleaned is to be achieved within the short time in which the textile is in the wash solution. Apparently the optical brighteners must first become solubilized in the aqueous wash solution and then deposit out on the textile. If the optical brightener is incorporated in the detergent composition in the form of large particles, the rate of solution is too slow and insufficient time is available in the washing cycle for the brightener to become solubilized in the wash solution and to deposit out on the textiles. In order to disperse these relatively water-insoluble brighteners in solid detergent compositions such as spray dried granules it has been suggested that the brightener be dissolved in such materials as alcohol prior to adding the brightener to the detergent slurry which is subsequently spray dried to form detergent granules. This process involves adding an extraneous material, i.e., the alcohol, to the detergent slurry, which in turn requires that the alcohol be subsequently removed during the spray drying. This method is wasteful.

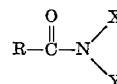
It is an object of this invention to provide a composition comprising a relatively water-insoluble optical brightener in solubilized form suitable for incorporation in detergent compositions.

It is a further object of this invention to provide an efficient process for incorporating relatively insoluble optical brighteners into detergent compositions.

These objects and other objects can be achieved by providing a composition consisting essentially of (1) a

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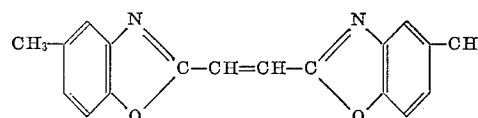
solvent selected from the group consisting of (a) an amide in liquid form having the formula



wherein



is an acyl group containing from about 8 to about 18 carbon atoms and X and Y are each selected from the group consisting of hydrogen, alkyl groups containing from one to about four carbon atoms, and hydroxy alkyl groups containing from one to about three carbon atoms and from one to about two hydroxy groups, (b) a fatty acid in liquid form containing from about 8 to about 31 carbon atoms, and (c) mixtures thereof, and (2) a brightener having the formula



said solvent having a temperature of less than about 250° F., said brightener being present in an amount greater than about 0.001% by weight of said solvent up to the limit of the brightener's solubility in the solvent and said brightener being essentially completely dissolved in said solvent.

The above composition can be utilized in the preparation of granular detergent compositions, especially anionic granular detergent compositions, e.g., by thoroughly mixing from about 0.001% to about 5% by weight, based on the finished granular detergent, of the said composition in an aqueous mixture which is then used to form the said granular detergent compositions by the removal of free moisture.

The brightener hereinbefore described is representative of relatively water-insoluble brighteners which have particular effectiveness, e.g., when incorporated in detergent compositions which are used to wash such synthetic textiles as "nylon" and "Dacron." The specific brightener hereinbefore mentioned, i.e., symmetrical 1,2-bis(5-methyl benzoxazolyl)-ethylene is particularly effective in brightening the aforesaid synthetic textiles as well as other textiles when incorporated into detergent compositions as hereinafter described.

The solvents in which the brightener of this invention are dissolved are useful and conventional components of detergent compositions.

Examples of suitable fatty amides include: ammonia amides; N-methyl amides; N-isopropanol amides; N-isobutyl amides; N-ethanol amides; N-diethanol amides; and mixtures of the preceding amides. The acyl groups in the above amides contain from about 8 to about 18 carbon atoms, i.e., caprylic, capric, lauric, myristic, palmitic and stearic acyl groups. Mixtures of chain lengths are suitable including acyl groups derived from natural oils such as coconut oil, palm oil, tallow, etc., and mixtures of acyl groups prepared synthetically. The acyl groups can be unsaturated as, for example, in oleamide. Further examples of suitable amides and their use in detergent com-

positions can be found in U.S. Patents 2,383,737 and 2,383,738. The choice of the specific amide used will depend upon the type of detergent composition desired and the availability of the particular amides. These amides are useful in enhancing the sudsing and detergency of synthetic detergent compositions.

Examples of suitable fatty acids and their use in detergent compositions include the fatty acid mixtures described in U.S. Patents 2,954,347 and 2,954,348. These mixtures of myristic, palmitic, stearic, arachidic, behenic, oleic, lignoceric, cerotic, melissic, and linoleic acids (i.e., chain lengths from about 14 to about 31 carbon atoms) are used as suds depressants, either alone or in combination with other suds depressants. Shorter chain length fatty acids such as those derived from coconut oil and including caprylic, capric, and lauric fatty acids can be used as solvents, but longer chain length fatty acids such as those derived from tallow or hydrogenated fish oils are preferred and are better solvents. The fatty acids can be neutralized in the finished product either wholly or in part to form a soap and thereby act as detergent surfactants and/or suds depressants.

The brightener hereinbefore described is incorporated in the solvents hereinbefore described while the solvents are in a liquid (molten) form. Suitable mixing action is provided to enable the brightener to dissolve in the solvents in a reasonable period of time. The incorporation of the brightener in the solvents is achieved relatively easily. From about 0.001% to the solubility limit, usually about 2% by weight, based on the solvent, preferably about 1% by weight, of the brightener is added to the solvents in molten form at a temperature which is less than about 250° F. The amount of brightener which can be incorporated in the solvents is related to the temperature of the amide. The higher the temperature the more brightener can be incorporated, but with higher temperatures the solvents undergo degradation reactions. In general, the solvents should be held at as low a temperature as possible while maintaining the solvents in a molten state but maintaining the temperature at a sufficiently high level to incorporate the desired amount of brightener.

The above solvent/brightener compositions can be incorporated in a detergent composition in an amount from about 0.001% to about 5% by weight of the detergent composition. Preferably from about .5% to about 3.0% by weight is used and, desirably, about 2% by weight of the solvent/brightener composition is used in the detergent composition. The solvent/brightener can be incorporated in the detergent composition by dispersing the solvent/brightener in the aqueous slurry of the composition which is spray dried, drum dried, or dehydrated by the addition of hydrate forming salts to form detergent granules. The solvent/brightener composition is normally added to the slurry-containing crutcher and is thereby thoroughly mixed into the granular detergent composition resulting from drying the slurry. By this method the brightener is dispersed in extremely finely divided form in the finished granular detergent composition. It is believed that the brightener in the finished detergent composition is in extremely finely divided form approximating a solution. When the brightener/solvent solution becomes an integral part of a granular detergent composition, it is present in either a solidified form or absorbed as a liquid in the solid granules at ordinary temperatures such as room temperatures, e.g., 60° F.-90° F.

When the detergent surfactant is essentially anionic in character and detergent granules are prepared by forming an aqueous slurry comprising an anionic surfactant as hereinbefore described, the solvent/brightener compositions of this invention possess particular utility. The brightener of this invention by itself is relatively insoluble in anionic detergent compounds and in aqueous anionic detergent slurries and consequently, with normal mixing, the brightener is not sufficiently dispersed in the granules, which are subsequently formed from the slurries when the

free water is removed, to effect substantial deposition of the brightener on the textiles during a short washing cycle. When the brightener is incorporated in anionic detergent compositions by the solvent/brightener solutions and process of this invention, the brightener is found to be much more effective during fabric laundering than when it is simply added to an anionic detergent-containing crutcher by itself. Usually anionic detergent slurries contain about 50% to about 70% solids, the balance being water.

The solvent/brightener solution can also be added to the detergent composition after the detergent composition is in solid form, e.g., by spraying the molten solvent/brightener solution onto the finished detergent particles or finished detergent briquettes. This latter approach is especially useful when the particular solvent employed is of the type which does not readily withstand the elevated temperatures associated with spray drying, e.g., highly unsaturated fatty acids, or when the detergent surfactant or other ingredients cannot be spray dried.

The amount of brightener in the finished detergent composition will range from about .001% to about .1% by weight of the detergent composition, preferably from about .01% to about .03% by weight and most preferably at .02% by weight of the composition.

The solvents of this invention containing the brighteners of this invention can be used in combination with a variety of detergent actives in forming detergent compositions. Suitable detergent surfactants which can be used in amounts from about 5% to about 50% by weight of the detergent composition in these detergent compositions include: synthetic organic detergents characterized by their high solubility in water, their resistance to precipitation by the constituents of hard water and their surface active and effective detergent properties, including:

Anionic synthetic detergents. This class of synthetic detergents can be broadly described as the water-soluble salts, particularly the alkali metal salts, of organic sulfuric reaction products having in the molecular structure an alkyl radical containing from about 8 to about 22 carbon atoms and a radical selected from the group consisting of sulfonic acid and sulfuric acid ester radicals. Important examples of the synthetic detergents which form a part of the preferred compositions of the present invention are the sodium or potassium alkyl sulfates, especially those obtained by sulfating the higher alcohols produced by reducing the glycerides of tallow or coconut oil; sodium or potassium alkyl benzene sulfonates, in which the alkyl group is a straight or branched chain containing from about 9 to about 15 carbon atoms, especially those of the types described in United States Letters Patent Nos. 2,220,099 and 2,477,383; sulfonated alpha olefins containing from about 12 to about 18 carbon atoms, sodium alkyl glyceryl ether sulfonates, especially those ethers of the higher alcohols derived from tallow and coconut oil; sodium coconut oil fatty acid monoglyceride sulfates and sulfonates; sodium or potassium salts of sulfuric acid esters of the reaction product of one mole of a higher fatty alcohol (e.g., tallow or coconut oil alcohols) and about three moles of ethylene oxide; sodium or potassium salts of alkyl phenol ethylene oxide ether sulfate with about four units of ethylene oxide per molecule and in which the alkyl radicals contain about 9 carbon atoms; the reaction product of fatty acids esterified with isethionic acid and neutralized with sodium hydroxide where, for example, the fatty acids are derived from coconut oil; sodium or potassium salts of fatty acid amide of a methyl taurine in which the fatty acids, for example, are derived from coconut oil; and others known in the art, a number being specifically set forth in United States Letters Patent Nos. 2,486,921, 2,486,922 and 2,396,278.

The alkyl groups contained in said detergent surfactants can be straight or branched and saturated or unsaturated as desired. The above list of detergent sur-

factants is exemplary and not limiting. Mixtures of the above detergent surfactants with themselves and with other detergent surfactants can be used. Sodium and potassium alkyl sulfates and/or alkyl benzene sulfonates as hereinbefore described are particularly desirable, especially in spray dried detergent granules. They can be used together in ratios of 3:1 to 1:3, preferably 55 parts sulfonate to 45 parts sulfate.

In addition to the above mentioned ingredients the detergent compositions can also contain from about 5% to about 90% by weight of the detergent composition of detergency builders. These detergency builders are preferably used in this percentage range in a sufficient amount to provide a ratio of detergency builder to detergent surfactant of from about 1:1 to about 8:1 on a weight basis. Examples of water-soluble inorganic alkaline detergency builder salts are alkali metal carbonates, phosphates, polyphosphates, and silicates. Specific examples of such salts are sodium and potassium tripolyphosphates, carbonates, pyrophosphates, phosphates, and hexametaphosphates. Examples of organic alkaline sequestrant builder salts are (1) alkali metal amino polycarboxylates [e.g., sodium and potassium ethylene diaminetetraacetates, N-(2-hydroxyethyl)-ethylenediamine, triacetates, nitrilotriacetates, and N-(2-hydroxyethyl)-nitrilotriacetates]; (2) alkali metal salts of phytic acid (e.g., sodium and potassium phytates—see U.S. Patent 2,739,942); (3) water-soluble salts of ethane-1-hydroxy-1,1-diphosphonate (e.g., the trisodium and tripotassium salts); (4) water-soluble salts of methylene diphosphonic acid (e.g., trisodium and tripotassium methylene diphosphonate and the other salts described in the copending application of Francis L. Diehl, Ser. No. 266,025, filed Mar. 18, 1963, now Patent No. 3,213,030); (5) water-soluble salts of substituted methylene diphosphonic acids (e.g., trisodium and tripotassium ethylidene, isopropylidene, benzylmethylidene, and halomethylidene diphosphonates and the other substituted methylene diphosphonates disclosed in the copending application of Clarence H. Roy, Ser. No. 266,055, filed Mar. 18, 1963, now Patent No. 3,422,021); (6) water-soluble salts of polycarboxylate polymers and copolymers as described in the copending application of Francis L. Diehl, Ser. No. 269,359, filed Apr. 1, 1963, now Patent No. 3,308,067 (e.g., polymers of itaconic acid, aconitic acid, maleic acid, mesaconic acid, fumaric acid, methylene malonic acid, and citronic acid and copolymers with themselves and other compatible monomers such as ethylene); and (7) mixtures thereof.

Mixtures of organic and/or inorganic builders can be used and are generally desirable. Especially preferred are the mixtures of builders disclosed in the copending application of Burton H. Gedge, Ser. No. 235,327, filed Nov. 5, 1962, now Patent No. 3,392,121, e.g., ternary mixtures of sodium tripolyphosphate, sodium nitrilotriacetate, and sodium ethane-1-hydroxy-1,1-diphosphonate.

Other minor ingredients can also be added, if desired, to the detergent compositions which are prepared using the amide-brightener composition of this invention. Such minor ingredients include soil suspending agents such as carboxymethylcellulose, sodium and potassium silicates, diluents such as sodium sulfate and sodium chloride, dye and benzotriazole. The total amount of these minor ingredients is preferably less than about 10% by weight of the detergent composition, but can be used in greater amounts if necessary to form the balance of the composition.

Normally the detergent surfactant, the builders and the minor ingredients are incorporated into the detergent granule but they can also be added individually in the form of particles.

All percentages, ratios, and parts herein are by weight unless otherwise specified.

The following examples demonstrate the desirable and unique compositions and processes of this invention.

EXAMPLE I

About 1% by weight, based on the solvent/brightener composition, of symmetrical 1,2(5-methylbenzoxazolyl) ethylene was dissolved in middle cut coconut N-monoethanol amide wherein the acyl groups in said amide had a distribution such that 2% were C₁₀, 66% were C₁₂, 23% were C₁₄ and 9% were C₁₆. The amide was held at a temperature of about 200° F. while the optical brightener was being dissolved with mixing in the amide. This amide-brightener composition was used in preparing a granular detergent composition having the following formula:

	Percent
Sodium tripolyphosphate	46.5
Sodium straight chain alkyl benzene sulfonate wherein the alkyl group distribution is about 10%—C ₁₀ ; 35%—C ₁₁ ; 25%—C ₁₂ ; 23%—C ₁₃ ; 2%—C ₁₄ ; and about 5% branched chain components which were not characterized as to chain length	17.5
Sodium toluene sulfonate	2
Sodium sulfate	15
Sodium silicate having a ratio of SiO ₂ :Na ₂ O of 1.6	5.9
Amide/brightener composition in the amount required to provide about 2.1% amide and about .02% optical brightener	2.1
Moisture	10

100 parts of the above ingredients were added to a crutcher in the proportions indicated and mixed together with an additional approximately 40 parts water. After the ingredients were thoroughly mixed together they were spray dried by conventional means into detergent granules of the above composition and moisture content. These detergent granules were especially effective in that the optical brightener was dispersed through said detergent granules in a highly dispersed form and, accordingly, the optical brightener was effectively deposited on synthetic textiles such as "nylon" and "Dacron" when the detergent granules were used to wash said textiles. The amide acted as a sudsing and detergency enhancing agent for the granular detergent.

When middle cut coconut or tallow N-diethanol, ammonia, N - methyl, N - dimethyl, N - ethyl, N-diethyl, N-propyl, N-diisopropyl, N-isobutyl, N-dibutyl, N-glycerol, N-diglycerol, N-methanol, and N-dimethanol amides or mixtures thereof are substituted either wholly or in part for the coconut N-monoethanol amide in the above example, substantially equivalent results are obtained in that the brightener is solubilized in the amide and is contained in the finished granular anionic detergent in a highly dispersed form and gives maximum deposition on washed fabrics.

When sodium or potassium alkyl sulfates derived from, e.g., tallow alcohols are substituted either wholly or in part (e.g., about 45%) for the sodium alkyl benzene sulfonate in the above example, substantially equivalent results are obtained in that the detergent granules contain the brightener in a highly dispersed form.

EXAMPLE II

About 1% by weight of the brightener of Example I is dissolved at about 200° F. in a mixture of fatty acids in liquid form consisting of 8% myristic acid, 29% palmitic acid, 18% stearic acid, 26% arachidic acid, 17% behenic acid, and 2% oleic acid to form a useful composition. Similarly tallow and/or coconut fatty acids can be substituted either wholly or in part for the above fatty acids to prepare useful compositions.

About 3 parts of the above fatty acid/brightener compositions are added, by spraying, to 97 parts of a granular mixtures of about 17% potassium alkyl (C₁₂ average) benzene sulfonate, about 43% sodium tripolyphosphate, about 6% sodium silicate solids (1.6:1 SiO₂:Na₂O),

and 18% of miscellaneous ingredients including water of hydration, sodium sulfate, etc., and about 16% sodium carbonate. The alkalinity of this composition converts added fatty acid into corresponding sodium soap, but without affecting the brightener or its finely dispersed character.

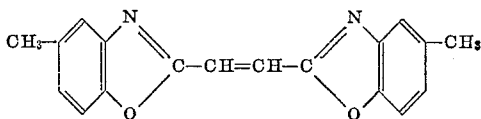
When the above ingredients, including the fatty acid/brightener compositions, are added to a detergent slurry in a crutcher with 40 parts of additional water, mixed together, and spray dried by conventional means to form detergent granules, essentially equivalent results are obtained in that the brightener is found in said detergent granules in a highly dispersed and particularly effective form.

The above detergent compositions are excellent for laundering and cleaning, e.g., textiles such as "nylon" and "Dacron" and contain brightener in a highly dispersed form which is effective in despositing upon and optically brightening said textiles.

What is claimed is:

1. The process of preparing a granular anionic detergent comprising the step of forming from about 0.001% to about 5% by weight, based on the finished granular detergent, of a mixture of

- (A) a fatty acid in liquid form containing from about 8 to 31 carbon atoms and
(B) a brightener having the formula



said brightener being present in an amount greater than about 0.001% by weight of said fatty acid sol-

vent up to the limit of said brightener's solubility in the fatty acid solvent and spraying said mixture at a temperature of less than 250° F. onto a granular mixture consisting essentially of

(C) an anionic synthetic detergent surfactant selected from the group consisting of sodium and potassium alkyl sulfates wherein the alkyl group contains 8 to 22 carbon atoms, alkylbenzenesulfonates wherein the alkyl group contains 9 to 15 carbon atoms and mixtures thereof, in an amount sufficient to give from about 5% to about 50% anionic synthetic detergent surfactant in the finished granular anionic detergent, and

(D) a water-soluble alkaline detergency builder material, the ratio of said detergency builder to said detergent surfactant being about 1:1 to about 8:1, whereby the alkalinity of the granular mixture converts fatty acid into the corresponding soap without affecting the brightener or its finely dispersed character.

2. The process of claim 1 wherein the granular mixture consists essentially of potassium alkylbenzenesulfonate and sodium tripolyphosphate.

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