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PROCESS OF BLEACHING SULFATED AND SULFONATED ANIONIC SYNTHETIC DETERGENT COMPOSITIONS AND THE RESULTING PRODUCTS

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This invention relates to improvements in bleached non-soap anionic synthetic detergent compositions and to a method of preparing such compositions which eliminates certain undesirable characteristics of these compositions, particularly as regards their tendency towards color reversion on standing. More particularly, the invention relates to such compositions that are of the sulfate or sulfonate type to be described more particularly hereinafter.

One of the disadvantages of present-day non-soap anionic synthetic detergent compositions is that the detergent composition after a brief period of time changes color. Although such a change in color does not in any way affect the effectiveness of the composition as a detergent, nevertheless the color change lessens its consumer appeal.

To overcome this disadvantage of non-soap anionic synthetic detergents, and especially those of the sulfate or sulfonate type, the present invention provides a new process for producing such detergent compositions in such condition that they will retain their original bleached color (and hence their original consumer appeal) over a greatly prolonged period.

Broadly, the invention provides a process of bleaching non-soap anionic synthetic detergent compositions in accordance with which an oxidizing agent which will quickly and effectively bleach the composition is added followed by the addition of another oxidizing agent which will prevent the regression of the bleaching effect obtained by the first oxidizing agent, thereby resulting in an improved detergent composition in which the tendency towards color reversion is eliminated or at least greatly minimized. More particularly, the present invention is based primarily on the feature of adding a hypohalite to an aqueous solution of a synthetic detergent of the kind indicated. followed by the addition of a peroxide to give a greatly improved non-soap anionic synthetic detergent composition, especially of the sulfate or sulfonate type.

It has been suggested heretofore to add hypohalites to synthetic detergent compositions for the purpose of bleaching same. However, when using hypohalites alone as the bleaching agent the bleaching effect is retained for only a relatively short period of time.

It is therefore an object of this invention to provide an improved non-soap anionic synthetic detergent composition which will retain its original color over a substantially prolonged period of time in comparison to an otherwise similar composition that has not been treated in accordance herewith.

Other objects and advantages of the present invention will become apparent upon consideration of the following detailed description:

According to the present invention it has been discovered that when a non-soap anionic surface active detergent (and particularly one of the sulfate or sulfonate type) is treated with a hypohalite followed by a peroxide, a new and improved synthetic detergent results which exhibits all of the desirable properties of the original detergent, such as good rinsing, foaming and deterging properties and, in addition, exhibits excellent color retention properties.

The non-soap anionic synthetic detergent compound, of the sulfate or sulfonate type, may be prepared from organic materials which are applicable for sulfonation ("true" sulfonation and/or sulfation), especially those having about eight to about twenty-six (and preferably about twleve to about twenty) carbon atoms to the molecule, including fatty oils, unsaturated fatty acids, mineral oils, mineral oil extracts, mono- and diglycerides, partial esters or ethers of polyglycols, esters or ethers of glycols, polyglycols and polyalcohols, aromatic and alkylated aromatic compounds, alcohols, and olefins, coal tar distillates, and numerous other organic compounds and mixtures of compounds. Such compounds can be sulfonated by any of several methods and may form any of several products, depending upon the method of sulfonation employed. The sulfonated or sulfated organic compounds include sulfonated mineral oil; conventional mineral oil refinery sludges; sulfonated mineral oil extracts; sulfonated fatty acids and oils; including sulfonated castor oil, sulforicinoleic acid, sulfonated olive oil, and sulfooleic acid, aliphatic sulfonates and sulfates, including cetyl sulfuric acid, dodecyl acid sulfate, and tetradecanehydroxy sulfonic acid-1,2; aliphatic ether and ester sulfonates including the dodecyl ether of hydroxy ethyl sulfonic acid, the cetyl ether of glyceryl sulfonic acid, tallow monoglyceride monosulfate, coconut oil monoglyceride monosulfate, hydrogenated tallow monoglyceride monosulfate, and hydrogenated coconut oil monoglyceride monosulfate; sulfonates prepared by treatment of organic materials with sulfur dioxide and chlorine in the presence of light followed by hydrolysis of the product; sulfonates prepared by treatment of organic bodies with sulfuryl chloride and an activating agent in the presence of light followed by the hydrolysis of the product; fatty acid amides of amino alkyl sulfonic acids, including lauric amide of taurine and tall oil acid amide of amino glyceryl sulfonic acid; sulfonic acids of naphthenes and naphthenic acids; lingnin sulfonic acids; aromatic and alkylated aromatic sulfonic acids, including naphthalene sulfonic acid, octadecyl benzene sulfonic acid, and dodecyl naphthalene sulfonic acid; the product of a mineral oil extract sulfonated while dissolved in liquid sulfur dioxide; and innumerable other organic sulfonic and sulfuric acid derivatives or mixtures thereof. These organic sulfate and true sulfonate compounds include those in which the organic radicals of the molecule are unsubstituted as well as those which contain substituents, such as halogens, halogenoids, hydroxyls, nitrogen-containing groups, acyl groups, acyloxy groups, alkoxy groups, etc.

More particularly, it has been discovered that when a water-soluble non-soap anionic synthetic detergent such as any of those mentioned above is treated with a hypochlorite such as sodium hypochlorite followed by a peroxide such as hydrogen peroxide, a greatly improved detergent composition results which exhibits all of the desirable properties of the original detergent and, in addition, exhibits color retention properties superior to any of the synthetic detergent compositions heretofore known. The detergent is preferably treated while in the form of a solution in a suitable solvent such as water and/or lower alcohol such as isopropanol.

In preparing the improved non-soap anionic synthetic detergent composition of this invention the hypohalite is preferably employed within the range of 0.5–1.5% and the peroxide should preferably be employed within the range of 0.25–0.9%, both based on the weight of the active synthetic detergent ingredient. The amount of peroxide added is at least sufficient to react with any residual hypochlorite; for example, according to the

following equation in the case of hydrogen peroxide and sodium hypochlorite:

$$H_2O_2+NaOCl \rightarrow H_2O+NaCl+O_2$$

In view of some possible uncertainty as to the amount of NaOCl actually consumed in the antecedent step, it is desirable that the H_2O_2 be at least chemically equivalent to the NaOCl added. This would correspond to the relationship: percent $H_2O_2=(0.46)\%$ NaOCl.

To further illustrate the general process of preparing the improved bleached non-soap anionic synthetic detergent compositions of the present invention, an evaporated aqueous alcoholic extract of a detergent composition (of which the active ingredient is sodium sulfate monoglyceride esters of fatty acids derived from hydrogenated coconut oil) is heated to a temperature of from 63° to 68° C. while stirring at a moderate rate. Thereupon, sodium hypochlorite is added while continuing to stir the composition. One half hour later hydrogen peroxide is added. After 1½ hours of stirring, a mixture of sodium sulfite and tetrasodium pyrophosphate is added. The solution is then stirred for 30 additional minutes. Thereafter the mixture may be spray-dried to form a powder which in turn may be extruded under pressure to form an improved non-soap anionic synthetic detergent bar product if desired.

Having described the present invention in its broader aspects, more detailed examples of procedures in accordance therewith are now given in order to assist in a better understanding of its various ramifications, it being understood that this is included herein for purpose of illustration rather than limitation. Percentages are by weight throughout the specification and claims unless otherwise indicated.

The following two examples relate to processes of preparing non-soap anionic synthetic detergent compositions to be made into powder and bar form, respectively.

EXAMPLE I

In the preparation of synthetic detergent composition in powder form, an aqueous alcoholic extract of the sodium salts of the sulfuric acid esters of the monoglycerides of the fatty acids derived from hydrogenated coconut oil is evaporated down to a solids content of approximately 37%. 673 grams of the resulting concentrate are heated in a one-liter, three-necked Morton flask fitted with a mechanical stirrer, a condenser, and a thermometer extending into the liquid. The solution is held at 63° to 68° C. (145° to 155° F.) throughout the process. Stirring is maintained at a moderate rate. When the solution reaches the designated temperature, 17.5 grams of a sodium hypochlorite solution (11% concentration, 7.0% on solids) are added. One half hour later 5.0 grams of hydrogen peroxide (30% concentration, 2.0% on solids) are added. After 1½ hours of stirring, a mixture of 5.0 grams of sodium sulfite (anhydrous, 2.0% on solids) and 1.25 grams of tetrasodium pyrophosphate (decahydrate, 0.5% on solids) is added. The solution is then spray-dried to powder form.

The sodium sulfite is added to decompose unreacted free hydrogen peroxide according to the equation:

$$Na_2SO_3+H_2O_2 \rightarrow H_2O+Na_2SO_4$$

The pyrophosphate is added to form a stable, colorless iron complex and thereby remove a small amount of color caused by the presence of dissolved iron salts.

The solution prior to spray drying is bright and clear. It gives a Lovibond reading (5¼ inch cell) of 1 Red and 4.6 Yellow and a pH (as is) of 7.0 compared to 4.5 Red, 28 Yellow and pH 6.8 for the starting material. The thus-treated synthetic detergent powder composition does not revert in color when maintained at a temperature of 55° to 60° C. over a period of two weeks.

EXAMPLE II

In the preparation of synthetic detergent composition for ultimate bar formation, 5900 lbs. of an evaporated aqueous alcoholic extract of the sodium salts of the sulfuric acid esters of the monoglycerides of the fatty acids derived from a mixture of hydrogenated coconut oil and hydrogenated tallow are placed in a tank. 161 lbs. of NaOCl (13% Cl_2) are added to the tank following which (after a lapse of 5–10 minutes) 40 lbs. of H_2O_2 (10%) are added with stirring. The temperature of the mixture is maintained at 150° F. (65.5° C.) throughout the oxidative treatment. After approximately one hour of stirring 7 lbs. of $Na_4P_2O_7$ are added to eliminate iron color interference and 33 lbs. of Na_2SO_3 are added to decompose unreacted free H_2O_2. The thus-bleached detergent composition is then sprayed into a spray-dryer. The resulting spray-dried granular material is then formed into detergent bars by forcing same through a plodder in otherwise conventional manner, to give a non-soap anionic synthetic detergent bar product of greatly enhanced color retention properties.

The following table shows the applicability of the process of the present invention to sill other non-soap anionic synthetic detergent materials. It indicates the Lovibond readings for an alkyl aryl sulfonate and an alcohol sulfate which are subjected to the same bleaching process as described above and thereafter maintained at a temperature of 50–60° C. for a period of three weeks:

Table

Detergent	Before Heating		After Heating	
	Bleached	Control (Unbleached)	Bleached	Control (Unbleached)
Alkyl Aryl Sulfonate [1]	11 R/110 Y..	50 R/140 Y..	13.8 R/138 Y..	54 R/140 Y.
Alcohol Sulfates [2]	1.4 R/14 Y..	6.3 R/63 Y..	2.0 R/20 Y....	(semi-solids).

[1] Sodium salt of benzene sulfonic acid alkylated with propylene tetramer.
[2] Sodium lauryl sulfate.

While the above examples have referred to the use of sodium hypochlorite and hydrogen peroxide which are the most common and readily available, it should be understood that any other suitable inorganic hypohalite and inorganic or organic peroxide may be used. For example, the alkaline earth metal hypohalites (such as calcium hypochlorite) and other alkali metal hypohalites (such as potassium hypochlorite) as well as the free acid itself (such as hypochlorous acid) may likewise be used with satisfactory results. All of these reagents are contemplated as falling within the scope of the expression inorganic hypohalite. Moreover, the sodium sulfite may be replaced by other alkali metal sulfites such as potassium sulfite and the tetrasodium pyrophosphate may be replaced by other phosphates including sodium tripolyphosphate and sodium hexametaphosphate as well as any of the corresponding potassium phosphates.

The time required for adequate bleaching is, generally speaking, a function of the temperature of operation, and both may vary over rather wide limits. In the particular working examples given above, the sulfated monoglycerides are bleached at temperatures of the order of 63° to 68° C. because of their tendency to hydrolyze at higher temperatures; however, more rapid treatment at higher temperatures is feasible with detergents which are stable at the given higher temperatures.

Summarizing, the present invention is based upon the discovery of a process of adding a hypohalite and a peroxide to a non-soap anionic synthetic detergent composition to form an improved synthetic detergent composition which will retain its original color over a greatly prolonged period of time.

While the invention has been described in terms of preferred embodiments thereof, it is to be understood that considerable variations in the details disclosed may be made without departing from the true scope and spirit of the invention. Accordingly, the invention is to be limited only by the claims set forth hereinafter.

What is claimed is:

1. A process of bleaching sulfated and sulfonated anionic synthetic detergents which comprises adding an inorganic hypohalite to a solution of such a detergent followed by adding a peroxide to said solution.

2. A process of bleaching sulfated and sulfonated anionic synthetic detergents which comprises adding an alkali metal hypochlorite to a solution of such a detergent followed by adding hydrogen peroxide to said solution to destroy excess ClO^-.

3. A process of bleaching sulfated and sulfonated anionic synthetic detergents which comprises adding an alkali metal hypohalite to a solution of such a detergent followed by adding a peroxide to said solution, said solution being maintained at an elevated temperature.

4. A process of bleaching sulfated and sulfonated anionic synthetic detergents which comprises adding sodium hypochlorite to a solution of such a detergent followed by adding hydrogen peroxide to said solution, said solution being maintained at a temperature of from about 63° to 68° C.

5. A process of bleaching sulfated and sulfonated anionic synthetic detergents which comprises adding from about 0.5 to 1.5% of an inorganic hypohalite to a solution of such a detergent followed by adding from about 0.25 to 0.9% of a peroxide to said solution.

6. A process of bleaching sulfated and sulfonated anionic synthetic detergents which comprises adding from about 0.5 to 1.5% of sodium hypochlorite to a solution of such a detergent while stirring followed by adding about 0.25 to 0.9% of hydrogen peroxide to said solution while stirring, said solution being maintained at an elevated temperature.

7. A process of bleaching a non-soap anionic synthetic detergent composition comprising alkali metal salts of sulfuric acid esters of monoglycerides of fatty acids derived from naturally-occurring fats and oils which comprises adding from about 0.5 to 1.5% of sodium hypochlorite to an aqueous solution of said composition while stirring followed by adding from about 0.25 to 0.9% of hydrogen peroxide to said solution while stirring, said solution being maintained at a temperature of from about 63° to 68° C. in the meantime.

8. A process of bleaching a non-soap anionic synthetic detergent composition comprising sodium salts of the sulfuric acid esters of the monoglycerides of the fatty acids from hydrogenated coconut oil which comprises adding from about 0.5 to 1.5% of sodium hypochlorite to a solution of said composition while stirring followed by adding from about 0.25 to 0.9% of hydrogen peroxide to said solution while stirring, said solution being maintained at a temperature of from 63° to 68° C. in the meantime.

9. A process of bleaching sulfated and sulfonated anionic synthetic detergents which comprises adding from about 0.5 to 1.5% of an inorganic hypohalite to an aqueous solution of such a detergent while stirring, adding from about 0.25 to 0.9% of a peroxide to said solution while stirring, and subsequently adding an alkali metal sulfite and an alkali metal phosphate to said solution.

10. A process of bleaching sulfated and sulfonated anionic synthetic detergents which comprises adding from about 0.5 to 1.5% of an inorganic hypohalite to a solution of such a detergent while stirring, adding from about 0.25 to 0.9% of a peroxide to said solution while stirring, and subsequently adding a mixture of sodium sulfite and tetrasodium pyrophosphate to said solution.

11. A bleached synthetic detergent composition as produced by the process of claim 1.

12. A bleached synthetic detergent composition as produced by the process of claim 9.

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