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㉝ Liquid detergent containing perborate bleach.

㉞ Stable, bleach-containing liquid detergents are prepared, by in situ crystallization of a perborate bleach. The crystallization may be carried out by converting perborate monohydrate to perborate tetrahydrate, or by converting a borate to a perborate.

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Liquid Detergent Containing Perborate Bleach

Technical Field

5 The present invention relates to aqueous liquid detergent compositions which contain perborate bleach in the form of small particles, i.e., particles having a weight average particle diameter of from 0.5 to 20 micrometers. The small particles are formed by in-situ crystallization, typically of perborate tetrahydrate. The tetrahydrate may be formed, e.g., by recrystallizing perborate monohydrate, or by reacting a borate with hydrogen peroxide.

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Background

15 So-called heavy duty liquid detergent compositions as are commercially available at present typically do not contain a bleach. Dissolved peroxygen compounds, like hydrogen peroxide, interact with other components generally used in liquid detergents, like enzymes and perfumes.

16 Insoluble peroxygen bleach compounds present the problem of poor physical stability of suspensions made therewith.

20 DE-OS 35 11 515, published October 17, 1985, discloses non-aqueous liquid detergent compositions comprising sodiumperborate monohydrate and an activator for the perborate. FR 2.579.615, published October 3, 1986, discloses similar non-aqueous compositions which further comprise catalase inhibitors. The compositions exemplified in these two patents do not contain anionic surfactants.

25 J. Dugua and B. Simon, "Crystallization of Sodium Perborate from Aqueous Solutions", Journal of Crystal Growth 44 (1978), 265-286, discusses the role of surfactants on the nucleation and crystal growth of sodium perborate tetrahydrate.

30 It is an object of the present invention to provide aqueous liquid detergent compositions containing perborate particles having a weight average particle diameter of from 0.5 micrometer to 20 micrometers. It is a further object of this invention to provide a process by which particles in the desired particle size range are formed in situ.

Summary of the Invention

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The aqueous liquid detergent compositions of the present invention have a pH of at least 8, comprise at least 5% of an organic non-soap anionic surfactant at least 5% of a builder, and from 1% to 40%, preferably from 10% to 20% of a perborate bleach in the form of particles having a weight average particle diameter of from 0.5 micrometer to 20 micrometers, said particles having been formed by in situ crystallization. Preferably, the perborate particles are formed by in situ crystallization of a perborate tetrahydrate, e.g. sodium perborate tetrahydrate.

40 Preferred liquid detergent compositions further comprise from 5% to 70% of a water-miscible organic solvent. The preferred water-miscible organic solvents are the low molecular weight monohydric alcohols; the most preferred of these solvents is ethanol.

45 Preferred herein are detergent compositions having a pH of at least 9, more preferably at least 9.5.

Detailed Description of the Invention

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The present invention addresses the problem of formulating an aqueous liquid detergent composition having suspended therein small particles of a perborate bleach. For reasons of physical stability it is necessary that the perborate particles have a weight average particle diameter of from 0.5 to 20 micrometers. It is not advisable to make such small particles by e.g., grinding, because this process is not very attractive economically. Moreover, such small particles in a dry state would pose serious industrial

hygiene and safety problems. It has also been found that detergent compositions containing small perborate particles that are obtained by grinding have poorer physical stability than compositions containing perborate particles of the same diameter that were obtained by *in situ* crystallization. Although this phenomenon is not fully understood, it is speculated that particle shape plays a role in it.

5 It has now been discovered that the required small perborate particles can be formed by *in situ* crystallization in the presence of at least 5% of an organic, non-soap, anionic surfactant and at least 5% of a detergent builder.

Percentages as used herein are percentages by weight of the liquid detergent composition. Weight percentages of the perborate are calculated as perborate monohydrate, even though the particles may be 10 different in composition (e.g., the tetrahydrate).

The term "in situ crystallization" relates to processes whereby perborate particles are formed from larger particles or from solution, in the presence of the water/anionic surfactant/detergent builder matrix. This term therefore encompasses processes involving chemical reactions, as when sodium perborate is formed by reacting stoichiometric amounts of hydrogen peroxide and sodium metaborate or borax. It also 15 encompasses processes involving dissolution and recrystallization, as in the dissolution of perborate monohydrate and subsequent formation of perborate tetrahydrate. Recrystallization may also take place by allowing perborate monohydrate to take up crystal water, whereby the monohydrate directly recrystallizes into the tetrahydrate, without dissolution step.

In one embodiment of the invention, a perborate compound, e.g., sodium perborate tetrahydrate or 20 sodium perborate monohydrate, is added to an aqueous liquid comprising the anionic surfactant and the detergent builder. The resulting slurry is stirred. During this stirring the perborate compound undergoes a process of dissolution/recrystallization. Due to the presence of the anionic surfactant and the detergent builder this dissolution/recrystallization process results in particles having the desired particle diameter.

As the monohydrate is more susceptible to recrystallization, the monohydrate is preferred for this 25 embodiment of the invention. Particle diameters herein are weight average particle diameters, unless otherwise specified. For reasons of physical stability it is preferred that the particle size distribution is relatively narrow; i.e., it is preferred that less than 10% of the perborate be present in the form of particles having a diameter greater than 25 micrometers, more preferably less than 10% (wt) has a particle diameter greater than 10 micrometers.

30 In a second embodiment of the invention the perborate compound is formed *in situ* by chemical reaction. For example, sodium metaborate is added to an aqueous liquid comprising the anionic surfactant and the detergent builder. Then a stoichiometric amount of hydrogen peroxide is added while stirring. Stirring is continued until the reaction is complete.

Instead of metaborate, other borate compounds, including e.g., borax and boric acid. If borax is used as 35 the boron compound, a stoichiometric amount of a base, e.g. sodium hydroxide, is added to ensure reaction of the borax to metaborate. The process then proceeds as described hereinabove for metaborate conversion. Instead of hydrogen peroxide, other peroxides may be used (e.g., sodium peroxide), as known in the art.

Normal, perborate tetrahydrate is formed. At temperatures above about 40 deg. C this may slowly 40 convert to the thermodynamically more stable trihydrate. This conversion may itself be used to make small particles of trihydrate out of large particles of tetrahydrate.

Preferred liquid detergent compositions contain, in addition to water, a water-miscible organic solvent. The solvent reduces the solubility of perborate in the liquid phase and thereby enhances the chemical stability of the composition.

45 It is not necessary that the organic solvent be fully miscible with water, provided that enough of the solvent mixes with the water of the composition to affect the solubility of the perborate compound in the liquid phase.

The water-miscible organic solvent must, of course, be compatible with the perborate compound at the 50 pH that is used. Therefore, polyalcohols having vicinal hydroxy groups (e.g. 1,2-propanediol and glycerol) are less desirable.

Examples of suitable water-miscible organic solvents include the lower aliphatic monoalcohols, and ethers of diethylene glycol and lower monoaliphatic monoalcohols. Preferred solvents are ethanol, isopropanol, 1-methoxy 2-propanol and butyldiglycolether.

Although the presence or absence of other ingredients plays a role, the amount of available oxygen in 55 solution is largely determined by the ratio water : organic solvent. The smaller this ratio (i.e. the more organic solvent is used in the solvent system), the lower the amount of available oxygen in solution. Although this is good for stability of the bleach system, it is less desirable for a good solubility of other components (e.g. electrolyte, anionic surfactants).

In practical terms, the ratio water : organic solvent is, for most systems, in the range from 8:1 to 1:3, preferably from 5:1 to 1:2. In calculating the amount of water in the detergent composition, allowances should be made for water released in or taken up by chemical and physical processes that may take place during the preparation of the detergent composition. For example, water may be formed in the neutralization 5 of an anionic surfactant, whereas water may be taken up in the conversion of metaborate to perborate tetrahydrate, as well as in the conversion of perborate monohydrate to perborate tetrahydrate. Water is also present in most detergent raw materials, and should be taken into account.

As it is believed that the ionic strength of the composition affects the crystallization process, preferred compositions have an ionic strength of at least 0.8 moles/liter, preferably from 2 to 3.5 moles/liter. Ionic 10 strengths are calculated on the assumption that all ionic materials other than perborate present in the composition are fully dissociated.

The liquid detergent compositions herein contain from 5% to 60% of the liquid detergent composition, preferably from 15% to 40%, of an organic surface-active agent selected from nonionic, anionic, and zwitterionic surface-active agents and mixtures thereof. At least 5% of the detergent composition must be 15 anionic surfactant.

Synthetic anionic surfactants can be represented by the general formula R₁SO₃M wherein R₁ represents a hydrocarbon group selected from the group consisting of straight or branched alkyl radicals containing from about 8 to about 24 carbon atoms and alkyl phenyl radicals containing from about 9 to about 15 carbon atoms in the alkyl group. M is a salt forming cation which typically is selected from the 20 group consisting of sodium, potassium, ammonium and mixtures thereof.

A preferred synthetic anionic surfactant is a water-soluble salt of an alkylbenzene sulfonic acid containing from 9 to 15 carbon atoms in the alkyl group. Another preferred synthetic anionic surfactant is a water-soluble salt of an alkyl sulfate or an alkyl polyethoxylate ether sulfate wherein the alkyl group contains from about 8 to about 24, preferably from about 10 to about 18 carbon atoms and there are from about 1 to 25 about 20, preferably from about 1 to about 12 ethoxy groups. Other suitable anionic surfactants are disclosed in U.S. Patent 4,170,565, Flesher et al., issued October 9, 1979.

The nonionic surfactants are conventionally produced by condensing ethylene oxide with a hydrocarbon having a reactive hydrogen atom, e.g., a hydroxyl, carboxyl, or amido group, in the presence of an acidic or basic catalyst, and include compounds having the general formula RA(CH₂CH₂)_nH wherein R represents 30 the hydrophobic moiety, A represents the group carrying the reactive hydrogen atom and n represents the average number of ethylene oxide moieties. R typically contains from about 8 to 22 carbon atoms. They can also be formed by the condensation of propylene oxide with a lower molecular weight compound. n usually varies from about 2 to about 24.

The hydrophobic moiety of the nonionic compound is preferably a primary or secondary, straight or 35 branched, aliphatic alcohol having from about 8 to about 24, preferably from about 12 to about 20 carbon atoms. A more complete disclosure of suitable nonionic surfactants can be found in U.S. Patent 4,111,855. Mixtures of nonionic surfactants can be desirable.

Zwitterionic surfactants include derivatives of aliphatic quaternary ammonium, phosphonium, and 40 sulfonium compounds in which the aliphatic moiety can be straight or branched chain and wherein one of the aliphatic substituents contains from about 8 to about 24 carbon atoms and another substituent contains, at least, an anionic water-solubilizing group. Particularly preferred zwitterionic materials are the ethoxylated ammonium sulfonates and sulfates disclosed in U.S. Patents 3,925,262, Laughlin et al., issued December 9, 1975 and 3,929,678, Laughlin et al., issued December 30, 1975.

Semi-polar nonionic surfactants include water-soluble amine oxides containing one alkyl or hydroxy 45 alkyl moiety of from about 8 to about 28 carbon atoms and two moieties selected from the group consisting of alkyl groups and hydroxy alkyl groups, containing from 1 to about 3 carbon atoms which can optionally be joined into ring structures.

Suitable anionic synthetic surface-active salts are selected from the group of sulfonates and sulfates. The like anionic detergents are well-known in the detergent arts and have found reexported application in 50 commercial detergents. Preferred anionic synthetic water-soluble sulfonate or sulfate salts have in their molecular structure an alkyl radical containing from about 8 to about 22 carbon atoms.

Examples of such preferred anionic surfactant salts are the reaction products obtained by sulfating C₈-C₁₈ fatty alcohols derived from tallow and coconut oil; alkylbenzene sulfonates wherein the alkyl group contains from about 9 to about 15 carbon atoms; sodium alkylglyceryl ether sulfonates; ether sulfates of 55 fatty alcohols derived from tallow and coconut oils; coconut fatty acid monoglyceride sulfonates and sulfonates; and water-soluble salts of paraffin sulfonates having from about 8 to about 22 carbon atoms in the alkyl chain. Sulfonated olefin surfactants as more fully described in e.g. U.S. Patent Specification 3,332,880 can also be used. The neutralizing cation for the anionic synthetic sulfonates and/or sulfates is

represented by conventional cations which are widely used in detergent technology such as sodium and potassium.

5 A particularly preferred anionic synthetic surfactant component herein is represented by the water-soluble salts of an alkylbenzene sulfonic acid, preferably sodium alkylbenzene sulfonic acid, preferably sodium alkylbenzene sulfonates having from about 10 to 13 carbon atoms in the alkyl group.

A particularly preferred anionic synthetic surfactant component herein is represented by the water-soluble salts of an alkylbenzene sulfonic acid, preferably sodium alkylbenzene sulfonates having from about 10 to 13 carbon atoms in the alkyl group.

10 A preferred class of nonionic ethoxylates is represented by the condensation product of a fatty alcohol having from 12 to 15 carbon atoms and from about 4 to 10 moles of ethylene oxide per mole of fatty alcohol. Suitable species of this class of ethoxylates include : the condensation product of C₁₂-C₁₅ oxo-alcohols and 7 or 9 moles of ethylene oxide per mole of alcohol; the condensation product of narrow cut C₁₄-C₁₅ oxo-alcohols and 7 or 9 moles of ethylene oxide per mole of fatty (oxo)alcohol; the condensation product of a narrow cut C₁₂-C₁₃ fatty(oxo)alcohol and 6,5 moles of ethylene oxide per mole of fatty alcohol; 15 and the condensation products of a C₁₀-C₁₄ coconut fatty alcohol with a degree of ethoxylation (moles EO/mole fatty alcohol) in the range from 5 to 8. The fatty oxo alcohols while mainly linear can have, depending upon the processing conditions and raw material olefins, a certain degree of branching, particularly short chain such as methyl branching.

20 A degree of branching in the range from 15% to 50% (weight%) is frequently found in commercial oxo alcohols.

25 Preferred nonionic ethoxylated components can also be represented by a mixture of 2 separately ethoxylated nonionic surfactants having a different degree of ethoxylation. For example, the nonionic ethoxylate surfactant containing from 3 to 7 moles of ethylene oxide per mole of hydrophobic moiety and a second ethoxylated species having from 8 to 14 moles of ethylene oxide per mole of hydrophobic moiety. A preferred nonionic ethoxylated mixture contains a lower ethoxylate which is the condensation product of a C₁₂-C₁₅ oxo-alcohol, with up to 50% (wt) branching, and from about 3 to 7 moles of ethylene oxide per mole of fatty oxo-alcohol, and a higher ethoxylate which is the condensation product of a C₁₆-C₁₉ oxo-alcohol with more than 50% (wt) branching and from about 8 to 14 moles of ethylene oxide per mole of branched oxo-alcohol.

30 The liquid detergent compositions herein optionally contain a fatty acid component. Preferred saturated fatty acids have from 10 to 16, more preferably 12 to 14 carbon atoms. Preferred unsaturated fatty acids are oleic acid and palmitoleic acid.

35 Detergent enzymes can be used in the liquid detergent compositions of this invention. In fact, one of the desirable features of the present compositions is that they are compatible with such detergent enzymes.

40 Suitable enzymes include the detergent proteases, amylases, lipases and cellulases. Enzymatic stabilizing agents for use in aqueous liquid detergents are well known. Preferred herein is a salt of formic acid, e.g., sodium formate. The amount of this stabilizing agent typically ranges from 0.5% to 2%.

Preferred compositions contain an inorganic or organic builder. Examples of inorganic builders include the phosphorous-based builders, e.g., sodium tripolyphosphate, sodium pyrophosphate, and aluminosilicates (zeolites).

45 Examples of organic builders are represented by polyacids such as citric acid, nitrilotriacetic acid, and mixtures of tartrate monosuccinate with tartrate disuccinate. Preferred builders for use herein are citric acid and alk(en)yl-substituted succinic acid compounds, wherein alk(en)yl contains from 10 to 16 carbon atoms. An example of this group of compounds is dodecetyl succinic acid. Polymeric carboxylate builders inclusive of polyacrylates, polyhydroxy acrylates and polyacrylates/polymaleates copolymers can also be used.

50 The compositions herein can contain a series of further optional ingredients which are mostly used in additive levels, usually below about 5%. Examples of the like additives include : polyacids, enzymes and enzymatic stabilizing agents, suds regulants, opacifiers, agents to improve the machine compatibility in relation to enamel-coated surfaces, bactericides, dyes, perfumes, brighteners and the like.

The liquid compositions herein can contain further additives of a level from 0.05 to 2%.

55 These additives include polyaminocarboxylates such as ethylenediaminetetraacetic acid, diethylenetriaminopentacetic acid, ethylenediamino disuccinic acid or the water-soluble alkali metals thereof. Other additives include organo-phosphonic acids; particularly preferred are ethylenediamino tetramethylene phosphonic acid, hexamethylenediamino tetramethylene phosphonic acid, diethylenetriaminopentamethylene phosphonic acid and aminotrimethylene phosphonic acid.

The compositions may further contain bleach stabilizers of the kinds known in the art. If a process involving the use of hydrogen peroxide is used for the preparation of the liquid detergent, typical bleach

stabilizers may be present as introduced with the commercially available hydrogen peroxide. Examples of suitable bleach stabilizers include ascorbic acid, dipicolinic acid, sodium stannates and 8-hydroxyquinoline can also be included in these compositions, at levels between 0.01 and 1%.

The beneficial utilization of the claimed compositions under various usage conditions can require the utilization of a suds regulant. While generally all detergent suds regulants can be utilized preferred for use herein are alkylated polysiloxanes such as dimethylpolysiloxane also frequently termed silicones. The silicones are frequently used in a level not exceeding 1.5%, most preferably between 0.1% and 1.0%.

It can also be desirable to utilize opacifiers inasmuch as they contribute to create a uniform appearance of the concentrated liquid detergent compositions. Examples of suitable opacifiers include : polystyrene commercially known as LYTRON 621 manufactured by MONSANTO CHEMICAL CORPORATION. The opacifiers are frequently used in an amount from 0.3% to 1.5%.

The liquid detergent compositions of this invention further can comprise an agent to improve the washing machine compatibility, particularly in relation to enamel-coated surfaces.

It can further be desirable to add from 0.1% to 5% of known antiredeposition and/or compatibilizing agents. Examples of the like additives include : sodium carboxymethylcellulose; hydroxy-C₁₋₆-alkylcellulose; polycarboxylic homo- or copolymeric ingredients, such as : polymaleic acid, a copolymer of maleic anhydride and methylvinylether in a molar ratio of 2:1 to 1:2; and a copolymer of an ethylenically unsaturated monocarboxylic acid monomer, having not more than 5, preferably 3 or 4 carbon atoms, for example (methyl)-acrylic acid, and an ethylenically unsaturated dicarboxylic acid monomer having not more than 6, preferably 4 carbon atoms, whereby the molar ratio of the monomers is in the range from 1:4 to 4:1, said copolymer being described in more detail in EUROPEAN Patent Application 0 066 915, filed May 17, 1982.

The following examples illustrate the invention and facilitates its understanding.

Liquid detergent compositions are prepared by mixing the listed ingredients in the stated proportions :

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| Ingredients | Composition (weight %) | | | | |
|--|------------------------|------|------|---------|------------------|
| | I | II | III | IV | V |
| Water | 33 | 32 | 26 | 23 | 34 |
| Ethanol | 14 | 15 | 18 | 22 | 11 |
| Linear dodecylbenzene sulfonic acid | 12 | 10 | 8 | 8 | 12 |
| Condensation product of 1 mole of C ₁₃ -C ₁₅ oxo alcohol and 7 moles of ethylene oxide | 7 | 9 | 10 | 8 | 7 |
| Sodium cocoyl sulfate | 2 | 3 | 4 | 2 | 2 |
| Dodecetyl succinic acid | 13 | 10 | 12 | 15 | 13 |
| Citric acid | 0.8 | 1 | 1 | 0.8 | 0.8 |
| Oleic acid | 3.3 | 4 | 3 | 2 | 3.3 |
| Protease | 0.3 | 0.5 | - | 0.5 | - |
| Diethylenetriamine pentamethylene phosphonic acid | 0.05 | 0.85 | 0.05 | 0.05 | 0.05 |
| Sodium formate | 0.9 | 1 | - | 1 | - |
| Sodium perborate monohydrate | 10 | 10 | 12 | 10 | 10 ^{a)} |
| Sodium hydroxide (to adjust to) pH | 9 | 10 | 9 | 11 | 8.2 |
| Perfume, minors | | | | balance | |

^{a)} sodium perborate tetrahydrate

50 The sodium perborate compound is added after all the other ingredients have been mixed. The composition is stirred overnight. The resulting recrystallized perborate particles have a weight average particle diameter of about 7 micrometers.

The following compositions are prepared in the same manner.

| Ingredients | Composition (weight %) | | | | | | |
|-------------------------------------|------------------------|------|------|------|---------|------|------|
| | VI | VII | VIII | IX | X | XI | XII |
| Water | 31 | 28 | 27 | 32 | 23 | 28 | 22 |
| Ethanol | 6.5 | - | 7 | 5 | 6 | 7 | 8 |
| 1-Methoxy-2-propanol | - | 14 | 3.5 | - | - | 7 | 7 |
| Isopropanol | 6.5 | - | - | - | 6 | - | - |
| Butyldiglycolether | - | - | 4.0 | 5 | - | - | - |
| Linear dodecylbenzene sulfonic acid | 11.4 | 12 | 7 | 13 | 12 | 10 | 9 |
| Nonionic surfactant | 7.2 | 7 | 11 | 3.5 | 6 | 8 | 7 |
| Sodium cocosulfate | 3.1 | 2.5 | 2.5 | 3.0 | 4.0 | 3.0 | 2.0 |
| TMS/TDS* | - | - | - | - | - | 6.5 | 3.5 |
| Dodecetyl succinic acid | 13.4 | 9 | - | - | 7 | 8.5 | 9.5 |
| Tetradecenylsuccinic acid | - | 4 | - | - | - | - | 1.0 |
| Coconut fatty acid | - | - | 16 | 1.0 | 1.0 | - | - |
| Oleic acid | 3.6 | 3.6 | 4.0 | 2.0 | 3.0 | 2.0 | 2.5 |
| Citric acid | 0.8 | 0.9 | 0.5 | - | 3.5 | - | 0.5 |
| DTPMPA** | 0.9 | 0.5 | 0.5 | - | 0.4 | 0.8 | 1.5 |
| Ethylene diamine tetraacetic acid | - | - | - | 1.0 | 0.4 | - | - |
| Sodium tripolyphosphate | - | - | - | 15.0 | - | - | - |
| Sodium perborate tetrahydrate | - | - | - | - | 17 | - | - |
| Sodium perborate monohydrate | 9.0 | 10.4 | 9 | 13 | - | 13 | 19 |
| Sodium formate | 0.9 | 0.8 | 1.0 | 1.0 | 1.5 | 0.5 | 1.0 |
| Protease | 0.7 | 0.6 | 0.8 | 0.5 | 1.0 | 0.5 | 0.6 |
| Sodium hydroxide to pH | 9 | 10 | 11 | 11 | 9.5 | 10.5 | 10.5 |
| Perfume, minors | | | | | balance | | |

* 80:20 mixture of tartrate monosuccinate and tartrate discuccinate

** Diethylenetriamino pentamethylene phosphonic acid

| Ingredients | Composition (weight %) | | | |
|-------------------------------|------------------------|--------|--------|--------|
| | XIII | XIV | XV | XVI |
| Water | 28 | 20 | 28 | 22 |
| Ethanol | 8 | 13 | 13 | 13 |
| Linear dodecylbenzene | 8.5 | 8.5 | 8.5 | 8.5 |
| nonionic surfactant | 7 | 7 | 7 | 7 |
| sodium cocosulfate | 1 | 1 | 1 | 1 |
| TMS/TDS | 12.5 | 12.5 | 12.5 | 12.5 |
| Citric Acid | 0.8 | 0.8 | 0.8 | 0.8 |
| Oleic Acid | 3.2 | 3.2 | 3.2 | 3.2 |
| Protease | 0.5 | 0.5 | 0.5 | 0.5 |
| DTMP | 0.85 | 0.85 | 0.85 | 0.85 |
| Formate | 0.9 | 0.9 | 0.9 | 0.9 |
| NaOH:to | 10 | 10 | 10 | 10 |
| H ₂ O ₂ | 16(30%) | 7(70%) | 7(70%) | 7(70%) |
| Metaborate.4H ₂ O | 20 | 20 | - | - |
| Borax | - | - | 13.5 | 13.5 |
| Minors | - | - | - | - |
| Total(parts) | 100 | 112 | 105 | 106 |
| Total Water | 42 | 24.5 | 28.5 | 22.5 |
| Ethanol | 8 | 13 | 13 | 13 |
| E/H | 0.19 | 0.53 | 0.46 | 0.58 |

Similar compositions are prepared as follows.

5 A liquid detergent matrix is prepared by mixing water, the organic solvent(s), the surfactant(s), and the builder material(s). The matrix is trimmed with sodium hydroxide to a pH of 8.5-9. Metaborate powder is added under stirring. A milky suspension is obtained. Then hydrogen peroxide is added as an aqueous solution. Small crystals of perborate tetrahydrate are formed. Typically, the perborate tetrahydrate crystals have a weight average particle size of about 4 micrometers.

As a result of the exothermic reaction the temperature rises to, typically, about 40 deg. C. The detergent composition is cooled to about 25 deg. C prior to addition of heat sensitive ingredients, like enzymes and perfume.

10 To the above detergent matrix, borax may be added in lieu of metaborate. The necessary amount of sodium hydroxide is added for the conversion of borax to metaborate. The metaborate is then converted to perborate through addition of hydrogen peroxide. After cooling to about 25 deg. C the heat sensitive components of the composition are added.

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Claims

1. An aqueous liquid detergent composition having a pH of at least 8, comprising :

a) at least 5% of an organic, non-soap anionic surfactant;

20 b) at least 5% of a builder; and

c) from 1%-40% of a perborate bleach in the form of particles having a weight average particle diameter of from 0.5 micrometer to 20 micrometers, said particles

25 2. A composition according to claim 1 wherein the perborate bleach particles have been formed by in situ reaction of a borate compound and a peroxide.

3. A composition according to claim 2 wherein the borate compound is metaborate or borax, and the peroxide is hydrogen peroxide.

4. A liquid detergent composition according to claim 1 wherein the perborate particles have been formed by recrystallization of a perborate monohydrate.

5. A liquid detergent composition according to any one of claims 1 through 4, further comprising from 30 5% to 70% of a water-miscible organic solvent.

6. A liquid detergent composition according to claim 5 wherein the water-miscible organic solvent is an aliphatic monoalcohol.

7. A liquid detergent composition according to claim 6, wherein the water-miscible organic solvent is ethanol.

35 8. A liquid detergent composition according to claim 7, wherein the solvent comprises water and ethanol in a water:ethanol ratio of from 8:1 to 1:3.

9. A liquid detergent composition according to claim 8, wherein the water:ethanol ratio is from 5:1 to 1:2.

40 10. A liquid detergent composition according to any one of the preceding claims having an ionic strength of at least 0.8 moles/liter.

11. A liquid detergent composition according to any one of the preceding claims having a pH of at least 9.

12. A liquid detergent composition according to claim 11 having a pH of at least 9.5.

45 13. A liquid detergent composition according to any one of the preceding claims containing less than 4% fatty acid.

14. A liquid detergent composition according to any one of the preceding claims comprising, from 5% to 40% of a builder selected from dodecetyl succinic acid; tetradecetyl succinic acid; dodecyl succinic acid; an 80:20 mixture of tartrate monosuccinate and tartrate disuccinate; citric acid; and mixtures thereof.

50 15. A process for preparing the liquid detergent compositions of any one of the preceding claims, comprising the steps of :

a) mixing water with the anionic detergent and the builder;

b) adding the perborate compound under stirring.

16. A process for preparing the liquid detergent compositions of any one of claim 1-14, comprising the steps of :

55 a) mixing water with the anionic detergent and the builder;

b) adding a borate compound;

c) adding a peroxide under stirring.

17. A process according to claim 16, wherein the borate compound is metaborate or borax, and the peroxide is hydrogen peroxide.

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