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(54) **Concentrated liquid detergent compositions.**

(57) The present invention relates to liquid detergent bleach-containing compositions which are formulated as a neat phase in order to exhibit good chemical and physical stability.

EP 0 619 368 A1

Technical Field

The invention relates to concentrated liquid detergent compositions which contain a suspending solid peroxygen compound.

Background

Answering the long-felt need for aqueous liquid detergent compositions containing a peroxygen bleach, which would be stable and efficient at the same time, EP-A-293 040 and EP-A-294 904 have described aqueous detergent compositions having a pH above 8 containing an anionic surfactant at conventional levels, i.e. above 5% by weight, typically from 15% to 40% by weight, and a solid water soluble peroxygen bleach, suspended in a specific water/solvent medium.

In such compositions, particular attention had to be given to the physical stability of suspended particles in the liquid medium. One option is represented by EP-A-0 430 330, describing liquid detergent compositions in which solid particles, in particular particles of a peroxygen compound, are suspended by means of a structured phase (surfactant "neat phase").

Surfactant structured (neat) phases are known as means to suspend solid particles such as zeolites in liquid detergent compositions, see in particular EP-A-79 646, EP-A- 170 091 and EP-A-295 021.

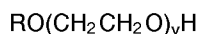
In bleach-containing liquid detergent compositions, chemical stability also represents an important concern, and high level of organic solvents should in principle be necessary, in order to achieve an optimal chemical stability. However, organic solvents at high levels have been found to have a detrimental effect on the formulation of surfactant neat phases, so that a good compromise between chemical and physical stability when using structured phases to suspend a peroxygen bleach compound was not easy to obtain.

The problem is especially acute in the case of percarbonate, which is more difficult to stabilize chemically than perborate.

The present invention provides concentrated liquid detergent compositions formulated as a neat phase in which solid suspended peroxygen compounds, such as perborate or percarbonate, in particular percarbonate, can be stabilized chemically and physically. Indeed, the present invention provides liquid detergent bleach-containing compositions which exhibit a very good chemical stability, and are in the form of a perfectly stable structured phase.

Summary of the invention

The present invention relates to a liquid detergent composition containing a surface-active agent, water, an organic solvent, and a solid peroxygen compound in the form of suspended particles. Said composition comprises at least 10% by weight of the total composition of a compound of the formula



wherein :

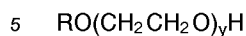
- R is a C_n alkyl, alkenyl, aryl or alkyl aryl group
- and n is an integer of from 4 to 22
- and y = 0 to 4
- and n/y ≥ 2 if y ≠ 0

In the composition according to the present invention the level of water is from 5% to 20% by weight of the total composition, and the weight ratio of organic solvent to water is of at least 0.8.

Detailed DescriptionThe structured phase

In the following, the expression "structured phase" refers to an essentially lamella-structured or spherulitic composition in which aqueous layers are separated by double layers of hydrophobic materials. Such a lamellar structure can be identified by observing the product through an optical microscope, between crossed Nichol prisms. The structured phases are characterized by specific textures which are described for instance by F.B. Rosevear, Journal of the American Oil Chemists Society, vol. 3, page 628, 1954.

Importantly, the compositions herein compulsory contain a surface-active agent and an organic solvent. Said compositions herein can be formulated as a structured phase by incorporation of at least 10% by weight of a compound of the formula



wherein

- R is a C_n alkyl, alkenyl, alkynyl, aryl or alkyl aryl group
- n is an integer of from 4 to 22
- 10 - y = 0 to 4
- and $n/y \geq 2$ if $y \neq 0$

or mixtures thereof.

Preferred compounds according to this definition are nonionic surfactants consisting of condensation products of ethylene oxide with a fatty alcohol in the presence of an acidic or basic catalyst. Fatty alcohols 15 having from 13 to 15 carbon atoms are preferably used to make such condensation products.

Also preferred compounds according to the above definition are such solvents as butanol, butoxyethanol, benzylalcohol, hexyl diglycol ether, butyl diglycolether, and mixtures thereof.

The preferred compositions herein therefore contain at least one of the organic solvents above, or a nonionic surfactant as defined above, or both types of compounds.

20 Most preferably, the organic solvents described above should be used in combination with another solvent such as described hereinafter. Similarly, the nonionic surfactants described above are preferably used in combination with an anionic surfactant, and/or other types of surfactants, including other nonionic surfactants, described hereinafter.

25 The water level and the water to organic solvent ratio

Two critical features of the present compositions are represented by the water level and the organic solvent to water weight ratio.

The water level is of 5% to 20% by weight of the present composition, i.e., the present compositions 30 are relatively concentrated.

The organic solvent to water weight ratio is at least 0.8, preferably at least 1.0.

It is the combination of these two features which allows to achieve both a good chemical and physical stability in the present liquid detergent compositions, in particular when the suspended peroxygen compound is percarbonate.

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The solid peroxygen compound

Suitable solid peroxygen compounds include the perborates, persulfates, peroxydisulfates, perphosphates and the crystalline peroxyhydrates formed by reacting hydrogen peroxide with sodium carbonate (forming percarbonate) or urea. Preferred peroxygen bleach compounds are perborates and 40 percarbonates. Percarbonates, known to be difficult to stabilize, have been found to be perfectly stabilized in the present composition, and are therefore the most preferred species for use herein.

The weight-average particle diameter of the peroxygen compounds herein must be in the range from 0.1 to 350 micrometers, preferably from 0.5 to 20 micrometers.

45 The required small particle size can be achieved by physical techniques, such as grinding, or any suitable chemical technique such as in-situ crystallization, especially for perborate, as it is described in detail in EP-A-294 904.

The peroxygen compounds are present in the compositions herein at levels of from 5% to 40%, preferably from 10% to 30%, most preferably from 15% to 25%, by weight of the composition.

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The organic solvent

The organic solvent preferably contains at least one solvent corresponding to the formula $\text{RO}(\text{CH}_2\text{CH}_2\text{O})_y\text{H}$ as described above, for the structured phase formation; butyl diglycol ether and hexyl 55 diglycol ether being preferred; other organic solvents which may be used preferably in combination with such required solvents, can be selected from the group of : polyalcohols having vicinal hydroxy groups (e.g. 1,2-propanediol and glycerol); lower monoaliphatic monoalcohols; specifically ethanol, n-propanol; isopropanol; polyethylene glycol (e.g., PEG 150, 200, 300, 400); dipropylene glycol; ethoxymethanol; methox-

yethanol; ethoxyethanol; ethyldiglycolether; benzylalcohol; butoxypropanol; butoxypropoxypropanol; and mixtures hereof.

Preferred are ethanol, propanediol, and mixtures thereof, when the peroxygen bleach compound is percarbonate, and ethanol when the peroxygen bleach compound is perborate.

5 The present liquid bleach compositions exhibit a pH (1% solution in distilled water) of at least 8, preferably of at least 9, more preferably of at least 9.5. The alkaline pH allows to get a good bleaching action of the peroxygen compound, particularly when the peroxygen is a percarbonate or perborate.

The surface active agent

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The compositions of the invention contain a surface active agent selected from anionic, nonionic, zwitterionic or cationic surface-active agents, and mixtures thereof.

Preferably the compositions herein contain an anionic, synthetic or natural surfactant.

15 Synthetic anionic surfactants can be represented by the general formula R^1SO^3M wherein R^1 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 group consisting of sodium, potassium, ammonium, and mixtures thereof.

20 Preferred among anionic synthetic surfactants are the water-soluble salts of alkylbenzene sulfonic acid, preferably sodium alkylbenzene sulfonates having from about 10 to 13 carbon atoms in the alkyl group.

Other suitable anionic surfactants can be represented by the water-soluble salts 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 20 carbon atoms, and preferably from about 1 to about 12 ethoxy groups for the alkyl polyethoxylate ether sulfates.

25 Particularly preferred are alkyl sulfates containing from 12 to 15 carbon atoms in the alkyl chain. Natural coconut or palm kernel alkyl sulfate are especially preferred.

Other suitable anionic surfactants are disclosed in U.S. Patent 4,170,565, Flesher et al., issued October 9, 1979.

30 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 $RA(CH_2CH_2O)_nH$ wherein R represents the hydrophobic moiety, A represents the group carrying the reactive hydrogen atom and n represents the average number of ethylene oxide moieties. n varies from about 4 to about 24.

35 R is an alkyl, alkenyl, aryl or alkyl aryl 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.

40 Zwitterionic surfactants include derivatives of aliphatic quaternary ammonium, phosphonium, and sulphonium 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.

45 Semi-polar nonionic surfactants include water-soluble amine oxides containing one alkyl or hydroxy 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.

50 Examples of suitable cationic surfactants include quaternary ammonium compounds of the formula $R_1R_2R_3R_4N^+X^-$, wherein R_1 is C_{12} - C_{20} alkyl or hydroxyalkyl; R_2 is C_1 - C_4 alkyl or C_{12} - C_{20} alkyl or hydroxyalkyl or C_1 - C_4 hydroxyalkyl, R_3 and R_4 are each C_1 - C_4 alkyl or hydroxyalkyl, or C_6 - C_8 aryl or alkylaryl; and X^- is halogen. Preferred are mono-long chain quaternary ammonium compounds (i.e., compounds of the above formula wherein R_2 is C_1 - C_4 alkyl or hydroxyalkyl).

The total amount of surfactant in the compositions is of from 5% to 60%, preferably 15% to 40% by weight of the total composition.

Optional ingredients

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The compositions of the invention may also contain a builder. Preferred are C_{10} - C_{16} alk(en)yl substituted succinic acids, typically present at levels of from 5% to 30% by weight of the total composition. The preferred builder for use herein is a mixture of dodecenyl substituted succinic acid and tetradecenyl

substituted succinic acid.

Examples of other suitable organic builders for use herein are represented by polyacids such as citric acid, nitrilotriacetic acid, and mixtures of tartrate monosuccinate with tartrate disuccinate. Polymeric carboxylate builders inclusive of polyacrylates, polyhydroxy acrylates and polyacrylates/polymaleates copolymers can also be used.

It is preferred, however, that those of the above builders which are water-soluble, be used at levels not exceeding 1% by weight of the total composition.

Other optionals include detergent enzymes. Suitable enzymes include the detergent proteases, amylases, lipases and cellulases. Enzymatic stabilizing agents for use in aqueous liquid detergents can also be used herein.

It is preferred, however, that salts of formic acid such as sodium formate, which is a typical enzyme stabilizing agent, be used at levels not exceeding 0.5% by weight of the total composition.

The compositions of the invention may also contain chelants, especially chelants for transition metal ions, since such ions can have a detrimental effect on the chemical stability of the compositions, the peroxygen bleaching agents being sensitive to these ions. Such chelants include ethylenediaminetetracetic acid, diethylenetriaminopentacetic acid, ethylenediamino disuccinic acid or the water-soluble alkali metals thereof. Other chelants include organophosphonic acids; particularly preferred are ethylenediamino tetramethylenephosphonic acid, hexamethylenediamino tetramethylenephosphonic acid, diethylenetriamino pentamethylenephosphonic acid, aminotrimethylenephosphonic acid and hydroxyethylidene 1,1 diphosphonic acid.

In the present context however, the very good chemical stability of the compositions makes it possible to include only very small levels of such chelants, typically 0.2%.

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

The beneficial utilization of the claimed compositions under various usage conditions can require the utilization of a suds regulant like polysiloxanes such as dimethylpolysiloxane, also frequently used in a level not exceeding 1.5%, most preferably between 0.1% and 1.0%

In selecting optional ingredients, it is also preferred that the level of electrolytes in the compositions herein is kept to a minimum. For instance, preferred compositions according to the present invention contain no more than 0.5% by weight of the total composition of added electrolytes such as sodium or potassium salts of bromide, chloride, iodide, nitrate and C1-4 alkyl carboxylates. Accordingly, the liquid detergent formulations of the present invention are substantially free of electrolytes.

Use of the compositions

The present concentrated compositions are designed to be used as is and are not diluted before use.

Example

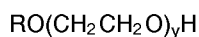
5	Ingredients	Composition wt%
	Linear alkyl benzene sulfonate	10
	C13-C15 alcohol ethoxylated (EO3)	10
	Dodecenyl/Tetradecenyl succinic acids*	19
10	Diethylenetriaminepentamethylene phosphonic acid	0.2
	Propanediol	5
	Ethanol	6
	Butyl diglycol ether	5
	Sodium Percarbonate	21
15	Protease	0.5
	Opacifier	0.6
	Dye (1%)	0.4
	Perfume	0.5
	Water	Up to 100
	Water content	13.9
20	Solvent content	16.5
	Solvent/Water content	1.18

*75% dodecenyl succinic acid, 25% tetradecenyl succinic acid

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Claims

1. A liquid detergent composition containing a surface-active agent, water, an organic solvent, and a solid peroxygen compound in the form of suspended particles, characterized in, that the composition comprises at least 10% by weight of the total composition of a compound of the formula



wherein :

- R is a C_n alkyl, alkenyl, aryl or alkyl aryl group
- and n is an integer of from 4 to 22
- and y = 0 to 4
- and n/y ≥ 2 if y ≠ 0

that the level of water is from 5% to 20% by weight of the total composition, and that the weight ratio of organic solvent to water is of at least 0.8.

2. A composition according to claim 1 wherein the compound of formula (ROCH₂CH₂O)_yH is selected from butyl diglycol ether, hexyl diglycol ether and the condensation products of C₁₃-C₁₅ fatty alcohols with 3 moles of ethylene oxide per mole of fatty alcohol, or mixtures thereof.
3. A composition according to claim 2 wherein the peroxygen compound is in the form of particles having a diameter size in the range of from 0.1 to 350 micrometers.
4. A composition according to claim 3 wherein the peroxygen compound is a percarbonate.
5. A composition according to claim 1, wherein the organic solvent contains ethanol or propanediol.
6. A composition according to claims 1 and 2, wherein the organic solvent is a mixture of ethanol or propanediol and butyldiglycol ether or hexyl diglycol ether.
7. A composition according to claims 1-6 wherein the surface active agent contains from 5% to 30% by weight of an anionic surfactant.

8. A composition according to claim 7 wherein the surface active agent contains a mixture of an anionic surfactant and a nonionic surfactant consisting of the condensation product of a C₁₃₋₁₅ fatty alcohol with 3 moles of ethylene oxide.
- 5 9. A composition according to claims 1-8, wherein it comprises from 5% to 30% by weight of the total composition of a C_{10-C₁₆} alk(en)yl substituted succinic acid.
- 10 10. A composition according to claims 1-9 characterized in that it contains no more than 0.5% by weight of the total composition of an added electrolyte such as sodium bromide, sodium chloride, sodium iodide, sodium nitrate, sodium acetate or sodium formate.

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EUROPEAN SEARCH REPORT

Application Number

EP 93 20 1008

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X,D	EP-A-0 430 330 (THE PROCTER & GAMBLE COMPANY) * claims 1-14; example X *	1-5,7-9	C11D17/00 C11D3/39
A	WO-A-9 112 309 (UNILEVER PLC) * page 5, line 10 - line 32; claims 1-8 *	1-10	
D,A	EP-A-0 293 040 (THE PROCTER & GAMBLE COMPANY) * the whole document *	1-10	
A	EP-A-0 385 522 (UNILEVER NV ET AL) * claims 1-14 *	1-10	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			C11D
The present search report has been drawn up for all claims			
Place of search BERLIN		Date of completion of the search 15 SEPTEMBER 1993	Examiner SIATOU E.
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			