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

(57) The invention pertains to stable built liquid detergent compositions comprising in an aqueous medium from 1.5% to 10% of an alkoxylated alcohol having less than 15 carbon atoms in the alkyl radical and an HLB between 11 and 13; from 2% to 22% of an anionic sulph(on)ate; up to 22% of an alkalimetal C₁₂-C₁₈ soap; from 10% to 30% of a builder and from 1% to 15% of an alkalimetal silicate. Compositions provide good stability under a wide range of storage conditions and increased formulating freedom in respect of the silicate component.

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AQUEOUS LIQUID DETERGENT COMPOSITIONS

This invention relates to built liquid detergent compositions. More particularly, the invention relates to aqueous built liquid detergent compositions with improved storage stability, which are particularly
5 suitable for semi-industrial laundering purposes.

Aqueous, built liquid detergent compositions for fabric washing are well known. They are often considered to be more convenient to the user than the equivalent dry
10 powdered or particulate products, as they are easier to dose, dissolve more readily, are non-dusting and usually occupy less storage space.

Their formulation, however, is difficult, since they
15 should be physically stable under a wide range of storage conditions, such as e.g. from about -5°C to $+37^{\circ}\text{C}$. They also should have satisfactory pouring properties notwithstanding high concentrations of detergent ingredients, especially builder salts necessary in
20 order to have a performance similar to that of the particulate detergent compositions.

In the prior art the problem of incorporating adequate quantities of e.g. inorganic builder salts in a liquid
25 detergent has been approached by providing liquid suspending media, which suspend the undissolved part of the builder salt in the liquid in a semi-permanent way.

Such a suspending liquid medium often comprises an
30 aqueous medium comprising an anionic surfactant, a non-ionic surfactant and an electrolyte. With such a ternary combination it is possible to produce a liquid medium capable of suspending adequate amounts of such undissolved or less soluble builders. Although the

suspending properties of systems of these types may be very good, hitherto it has proven difficult to formulate compositions having also satisfactory wide temperature range stability and viscosity.

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Accordingly, it is an object of the present invention to provide built liquid detergent compositions of the suspension type having an improved storage stability over a wide range of temperatures.

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It is often desirable to include in such detergent compositions certain amounts of silicates, such as e.g. sodium metasilicate, which serve as a source of alkalinity, have a buffering capacity and which inhibit corrosion.

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It has been found that such inclusion often suffers from two drawbacks. Firstly, the concentration range of silicate which can be incorporated without causing instability problems, is very narrow, thus providing poor formulating freedom. Secondly, and more important, the concentration range of silicate corresponding to stable products at one temperature, often overlaps very poorly or does not overlap at all with the concentration range corresponding to stable products at another temperature. Thus products which are stable at -4°C may not be stable at the same silicate level at 37°C or vice versa.

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Accordingly, it is a further object of the present invention to provide compositions containing a concentration range of silicate within which products are obtained which are stable over a wide range of storage conditions.

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It has now been found that built liquid detergent compositions which are stable over a wide range of storage

conditions and which comprise considerable levels of detergency builders and alkali metal silicate, can be formulated by using a ternary detergent-active system comprising an anionic synthetic detergent, a soap and a
5 nonionic synthetic detergent, in which the latter is an alkylene oxide condensate of an aliphatic alcohol having less than 15 carbon atoms in its alkyl radical and having a hydrophilic-lipophilic balance of between 11 and 13. By the invention, compositions are obtained
10 which remain stable for at least two months at -5°C , room temperature and $+37^{\circ}\text{C}$. Another suitable means for assessing the stability is the so-called freeze-thaw stability test, in which the product is cycled between -5°C and $+20^{\circ}\text{C}$ at a rate of one cycle per 24 hours.
15 Compositions of the invention remain stable even after at least six 24-hour cycles.

Accordingly, the present invention provides a stable built liquid detergent composition which comprises, in
20 an aqueous medium:

- (a) from 1.5% to 10% by weight of the total composition of a nonionic detergent which is an alkylene oxide condensate of aliphatic alcohol having less than 15 carbon atoms in its alkyl radical and having a
25 hydrophilic-lipophilic balance of between 11 and 13;
- (b) from 2% to 22% by weight of the total composition of a water-soluble anionic sulphate or sulphonate detergent;
- (c) up to 22% by weight of the total composition of
30 an alkali metal soap of saturated or unsaturated fatty acids having 12 to 18 carbon atoms;
- (d) from 10% to 30% by weight of a detergency builder; and
- (e) from 1% to 15% by weight of an alkali metal silicate.
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An essential feature of the present invention is the

- inclusion in the detergent-active surfactant mixture of a nonionic detergent which is an alkylene oxide condensate of an aliphatic alcohol having less than 15 carbon atoms in its alkyl radical and having an HLB of
5 between 11 and 13. The nonionic detergent is normally present in an amount of 1.5% to 10%, preferably 2% to 6% by weight of the total composition. Suitable non-ionic detergents are the condensation products of ethylene oxide, propylene oxide and/or butylene oxide with
10 aliphatic alcohols having less than 15 carbon atoms, preferably between 9 and 13 carbon atoms, C_{13} included, and most preferably 12 and 13 carbon atoms. The HLB, which may vary between 11 and 13, determines, in combination with the alcohol chain length, the suitable
15 number of alkylene oxide units. An example of a suitable nonionic is the product sold under the trade-name Dobanol 23-6.5 ex Shell Chem. Ltd (C_{12} - C_{13} alcohol consensed with 6.5 moles of ethylene oxide).
- 20 The water-soluble anionic sulphate or sulphonate detergent is normally present in an amount of 2% to 22%, preferably 2% to 10% or even 2 to 6% by weight of the total composition. The water-soluble anionic sulphonate detergents usable in the composition of the invention
25 are, for example, the alkali metal salts of C_{10} - C_{16} alkyl benzene sulphonates, C_{10} - C_{20} alkane sulphonates and C_{10} - C_{20} olefin sulphonates, the alkali metal salts of alkyl benzene sulphonates being preferred, especially those derived from alkylbenzenes having a C_{10} - C_{14} alkyl
30 chain length and an average molecular weight of approximately 225-245.

The water-soluble anionic sulphate detergents usable in the composition of the invention are primary and
35 secondary alkyl sulphates having an alkyl chain length of about 8 to 20 carbon atoms, preferably 12 to 18 carbon atoms, e.g. lauryl sulphate, and alkyl ether sul-

phates having 1-10 ethylene and/or propylene oxide units and having an alkyl chain length of about 8 to 20 carbon atoms, preferably 12 to 18 carbon atoms.

- 5 The alkali metal fatty acid soaps are normally present in an amount of up to 22%, preferably 2% to 22% and most preferably 2% to 6% by weight of the total composition.

- Typical examples of suitable soaps are those derived
10 from fatty acids having 12 to 18 carbon atoms, such as oleic acid, ricinoleic acid, and fatty acids derived from castor oil, rapeseed oil, groundnut oil, coconut oil, palm kernel oil or mixtures thereof. The sodium or potassium soaps of these acids can be used, the sodium
15 soaps being preferred.

- The compositions of the present invention contain 10% to 30%, preferably 10% to 25% by weight of the total composition of a detergency builder. Typical examples
20 of suitable detergency builders are the phosphate builders such as the alkali metal salts of tripolyphosphoric acid, pyrophosphoric acid, orthophosphoric acid, hexametaphosphoric acid and mixtures thereof. Sodium tripolyphosphate is preferred. Other suitable
25 builders include carbonates, zeolites and organic builders such as citrates, and polycarboxylates such as nitrilotriacetate.

- The silicate may be present in an amount of from 1% to
30 15%, preferably from 3% to 10% by weight of the total composition. Examples of suitable silicates are the alkali metal orthosilicates, metasilicates and disilicates, sodium metasilicate being preferred.

- 35 The liquid detergent composition of the invention may further contain any of the adjuvants normally used in fabric-washing detergent compositions, e.g. sequestering

agents such as ethylene-diaminetetraacetate and diethylene tetraamine methylene phosphoric acid; soil-suspending and anti-redeposition agents such as sodium carboxymethylcellulose, polyvinylpyrrolidone and the
5 maleic anhydride/vinylmethylether copolymer; fluorescent agents; hydrotropes; lather boosters; perfumes, germicides and colourants.

Further, the addition of lather depressors such as
10 liquid polysiloxane anti-foam compounds; enzymes, particularly proteolytic and amylolytic enzymes; peroxygen or chlorine bleaches, such as sodium sulphite, sodium perborate and potassium dichlorocyanurate, including
bleach activators, such as N,N,N',N'-tetraacetyl-
15 ethylene diamine, may be necessary or desirable to formulate a complete heavy-duty detergent composition suitable for use in machine washing operations. These ingredients can be employed in the liquid detergent compositions of the invention without danger of undue
20 decomposition during storage, especially if a proper protective coating is applied.

Compositions of the present invention normally have viscosities within the range of 0.5 to 3.0 Pas (at 20°C
25 and 20 sec⁻¹), preferably within the range of 0.8-1.5 Pas.

Depending on the specific use, compositions may be formulated having pH values within the whole alkaline
30 region, but preferred are pH values above 12 or even above 13, such as e.g. from 13.2 to 13.7.

The invention will be further illustrated in the following examples. All percentages used herein are by
35 weight unless otherwise specified.

The compositions of Examples 1 to 13 were prepared according to the formulations shown in Table I, all being stable homogeneous and pourable products.

- 5 The stability, assessed both on storage (2 months at -5°C , $+22^{\circ}\text{C}$ and 37°C) and on freeze/thaw cycling (10 cycles of 24 h/24 h at $-5^{\circ}\text{C}/+22^{\circ}\text{C}$), was excellent for all compositions.

TABLE I

Ingredients	Examples (amounts in %)												
	1	2	3	4	5	6	7	8	9	10	11	12	13
C ₁₂ -alkyl benzene-sulphonic acid (98%)	6.5	6.2	5.2	3.2	7.5	10.5	11.3	5.2	6.2	16.0	10.6	10.6	6.2
C ₁₂ -C ₁₃ alcohol condensed with 6.5 moles of ethyl-oxide (HLB = 11.9)	3.2	3.6	3.4	3.9	4.5	3.0	3.7	3.9	3.6	4.6	7.8	7.8	3.6
Oleic acid	3.3	3.2	3.3	5.9	3.0	1.5	-	3.9	3.0	2.3	6.6	6.6	3.2
Sodium tripolyphosphate	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	22.0	12.0	10.0	10.0	20.0
Sodium metasilicate	5.0	5.0	5.0	7.0	4.0	4.0	4.0	5.0	5.0	4.0	4.0	6.0	9.5**
Sodium hydroxide (34%)	3.2	3.3	3.3	3.2	3.6	4.1	3.9	3.2	3.3	6.2	6.4	6.4	9.9
Fluorescer *	-	0.3	0.2	0.3	-	-	-	0.2	0.3	-	-	-	-
Water	58.6	58.4	59.6	56.5	57.4	56.9	57.1	58.6	56.4	54.9	54.6	52.6	47.6
pH				13.3	13.6	13.4	13.6	13.3	13.5	13.4	13.2	13.7	14.0
Viscosity in Pa.s at 22°C, 21 sec ⁻¹ , - imm. after prepar. - after 2 months				0.5	0.9	1.4	1.5	1.2	0.5	1.9	0.8	0.7	
				0.9	1.2	1.3	1.1	1.0	1.3	2.1	2.7	0.8	

* Tinopal CBS-X (distyrylbiphenyl derivative ex Ciba Geigy) or Blankophor BHC (diaryltriazole stilbene derivative ex Bayer)

** 34% solution of sodium disilicate

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The compositions of Examples 14 and 15 were prepared according to the formulations in Table II, giving stable, (both on storage and cycling) homogenous and pourable products.

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TABLE II

	Ingredients	Examples	
		14	15
10	C ₁₂ -alkylbenzene sulphonic acid (98%)	6.5	5.8
	C ₉ -C ₁₁ alcohol condensed with 6 moles of ethylene oxide	3.2	3.9
15	(HLB = 12.0)		
	Oleic acid	3.3	3.3
	Sodium tripolyphosphate	20.0	20.0
	Sodium metasilicate	5.0	5.0
	Sodium hydroxide (34%)	3.4	3.2
20	Fluorescer (Tinopal CBS-X)	0.2	0.2
	Water	58.4	58.3
	pH	13.7	13.7
	Viscosity in Pa.s (immediately after preparation)	1.0	0.7
25	(after 2 months)	1.1	1.0

For reasons of comparison Examples 16 to 18 in Table III illustrate stability problems if commercially available nonionic products are used which fall outside the definition of the present invention.

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TABLE III

Ingredients	Examples		
	16	17	18
C ₁₂ -alkylbenzene sulphonic acid (98%)	6.2	6.2	6.2
Synperonic A7 1) (HLB = 12.2)	3.6	-	-
Dobanol 25-7 2) (HLB = 12.0)	-	3.6	-
Synperonic 87K 3) (HLB = 12.0)	-	-	3.6
Oleic acid	3.2	3.2	3.2
Sodium tripolyphosphate	20.0	20.0	20.0
Sodium metasilicate	5.0	5.0	5.0
Sodium hydroxide (34%)	3.3	3.3	3.3
Water	58.7	58.7	58.7
Stability after 2 months' storage			
- at 37°C	-	-	+
- at 22°C	+	+	-
- at -5°C	+	-	-
Stability on cycling (10 x 24 h/24 h at -5°C/22°C)	+	-	-

- 1) C₁₂-C₁₅ alcohol condensed with 7 moles of ethylene oxide, ex ICI
- 2) C₁₂-C₁₅ alcohol condensed with 7 moles of ethylene oxide, ex Shell
- 3) C₁₂-C₁₅ alcohol condensed with 7 moles of ethylene oxide, ex ICI

CLAIMS

1. A stable built liquid detergent composition including in an aqueous medium a nonionic detergent, an anionic detergent and a soap, characterized in that it comprises:
- 5 (a) from 1.5% to 10% by weight of a nonionic detergent which is an alkylene oxide condensate of an aliphatic alcohol having less than 15 carbon atoms in its alkyl radical and a hydrophilic-lipophilic balance of between 11 and 13;
- 10 (b) from 2% to 22% by weight of a water-soluble anionic sulphate or sulphonate detergent;
- (c) up to 22% by weight of an alkalimetal soap of saturated or unsaturated fatty acids having 12 to 18 carbon atoms;
- 15 (d) from 10% to 30% by weight of a detergency builder; and
- (e) from 1% to 15% by weight of an alkalimetal silicate.
- 20 2. A composition according to claim 1, characterized in that it comprises from 2% to 6% of the nonionic detergent.
3. A composition according to claim 1 or 2, characterized in that the nonionic detergent is derived from
- 25 an aliphatic alcohol having between 9 and 13 carbon atoms.
4. A composition according to any preceding claim, characterized in that it comprises from 2% to 6% by
- 30 weight of the anionic detergent.
5. A composition according to any preceding claim, characterized in that it comprises from 2% to 6% by
- 35 weight of the soap.

6. A composition according to any preceding claim, characterized in that it comprises from 10% to 25% by weight of the builder.

5 7. A composition according to any preceding claim, characterized in that it comprises from 3% to 10% by weight of the silicate.

8. A composition according to any preceding claim,
10 characterized in that the silicate is sodium meta-silicate.

9. A composition according to any preceding claim, characterized in that it has a pH value above 13.



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EUROPEAN SEARCH REPORT

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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. 3)
X	EP-A-0 038 101 (UNILEVER N.V.) * Pages 6, 7; examples 1-5; claim 1 *	1,2,4,5,6,8	C 11 D 1/83 C 11 D 10/04
X	DE-A-2 447 945 (UNILEVER N.V.) * Page 13, example 6 *	1-3,5-7	
			TECHNICAL FIELDS SEARCHED (Int. Cl. 3)
			C 11 D 1/00 C 11 D 10/00
The present search report has been drawn up for all claims			
Place of search BERLIN		Date of completion of the search 22-11-1983	Examiner SCHULTZE D
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	