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A request for correction of some errors in the description and in the claims has been filed pursuant to Rule 88 EPC. A decision on the request will be taken during the proceedings before the Examining Division (Guidelines for Examination in the EPO, A-V, 2.2).

The title of the invention has been amended (Guidelines for Examination in the EPO, A-III, 7.3).

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I-16124 Genova(IT)(54) **Dilutable concentrated detergent composition.**

(57) A composition comprising a mixture of anionic surfactants, of the type alkylaryl sulfonates, alkansulfonates, alkylethoxysulfonates, non-ionic surfactants, of the type oxyethylene polycondensates of alkanolamides of fatty acids, alcohols, alkylphenols, and solvents of the type alcohols, glycols, glycol-ethers and ketones having a low molecular weight. The ratio of anionic to non ionic surfactants in the said composition is comprises in the range between 10:1 to 1:1, with a total amount of active matter ranging from 60 to 85%. The composition is dissolved in water at the ambient temperature in a time ranging from 3 to 5 minutes by simple manual shaking, providing a ready to use liquid detergent with a pH between 6.5 to 7.5.

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-Concentrated liquid detergent composition adapted for preparing liquid light duty laundry or dishwashing detergents-

This invention relates to liquid detergents, and more particularly to a liquid light duty detergent composition complete with all the ingredients, including the minor components, having a high concentration of active matter, in the form of a viscous liquid, quickly dilutable in water, suitable for the preparation, from prefixed monodoses of concentrated product, of liquid detergents ready for use.

5 Detergents, and more particularly liquid detergents, are substantially formed by surfactants, either of anionic, non-ionic or amphoteric type, defined as active agents, and by various materials added to obtain particular effects, such as for example bleaching agents or builders in case of laundering products, or abrasive powders in case of bathroom or kitchen-cleaning products, and/or minor agents such as electrolytes, perfumes, preservatives, dyes, and the like.

10 Among the anionic surfactants, the most widely used is the straight chain alkylaryl sulfonate, in the form of sodium, potassium, ammonium and alkanol ammonium salts (LAS Na, LAS K, Las Tea), or a combination of the above. In addition to alkylaryl sulfonates, also sodium alkanesulfonates (SAS Na), sulfonated alpha-olefines, and sodium alkylethoxy sulfates (AES Na) with low etoxylation degree are frequently used.

Among the non-ionic surfactants, the most widely used are the mono- and di-alkanolamides, the
15 ethylene oxide condensates on alkanolamides, or fatty alcohols, or fatty acids, or alkylphenols.

Among the amphoteric surfactants, the betaine derivatives, imidazole derivatives, and amine oxides are widely used.

Concentrated detergent compositions, based on mixtures of anionic, non-ionic and amphoteric surfactants, are known.

20 So, for instance, in the U.S. patent No. 3,574,125 a process is described according to which to a concentrate, consisting essentially of an anionic aliphatic capillary-active sulfate or carboxylate C 12 -C 14, a suitable plasticizer is added, which promotes the dissolving rate in water of the anionic detergent concentrate. However, the above composition is intended for use in factory, for the mass production of diluted liquid detergents, and not for the extemporary household production of little amounts of diluted
25 detergents, without the aid of mechanical stirring equipments. Infact, we have made experiments with the composition cited in Examples 10, 11 and 12 of the above patent No. 3,574,125 and found that the solution of the above composition by simply manual shaking of the dissolving container took place after more than 60 minutes.

In the U.S. patent No. 3,709,838 a polifunctional essentially 100% active stable liquid detergent
30 composition is described, which may be diluted to commercially acceptable concentrations. Also in this case, however the dilution must be performed at industrial level. Infact, we have made experiments with the compositions cited in Examples 6 and 7 of said patent and found that the solution of the above composition by simply manual shaking of the dissolving container took place after a time varying from 30 to 60 minutes, that means a time which is not acceptable for the purposes of the present invention.

35 The U.S. patents Nos. 4,079,078 and 4,105,152 as well the U.K. patent No. 1,527,141 are all concerned with heavyduty laundry detergents, in which the ratio between non-ionic and anionic surfactants is comprised between 5:1 and 2.5:1, which renders the said detergent compositions fully unsuitable for the use as dishwashing detergents. Moreover, the detergent compositions disclosed in the above patents contains a certain amount of free ethanolamine in order to confer an alkaline pH to the washing solution.

40 We have made experiments on the composition of Example 1 of the U.S. patent No. 4,105,592, which may be considered as representative of the detergent compositions of the above mentioned prior art patents.

By diluting with water the detergent composition of the above mentioned Example 1 to the desired concentration of use, the same was found to have a fully satisfactory dissolving rate.

45 However, by modifying the composition of the above mentioned prior art patent so as to obtain: a) a concentrate containing about 70% of active matter; b) by reducing the non-ionic surfactants content in the composition and c) by eliminating the free alkali in the composition so as to obtain a neutral pH value, as highly desirable especially for a light duty and dishwashing detergent, a dissolving time of more than 60 minutes was obtained.

50 From the Japanese Patent No. 0079904 a liquid detergent composition of high concentration is known, containing sulphuric acid ester salt of alcohol ethoxylate, sec. alcohol ethoxylate, alkane sulphonate and ethanol. However, also this patent is not concerned with a composition to be diluted by a simple manual shaking of the dissolving container, within a reasonably short time, as requested by a product which is

intended for household use. Also in this case, the concentrated product of this composition is intended for industrial use. Moreover, we have made experiments on the product of the above Japanese Patent, and found that by diluting the disclosed composition at a dilution of active matter of 12%, same forms a gel, which is unsuitable for the purposes for which the composition of our invention is intended.

5 It is therefore the main object of this invention to provide a highly concentrated detergent composition suitable for the preparation of light duty dishwashing or laundry detergents having an active matter content from 60 to 85%, which is readily soluble in water, with dissolving rates from 1 to 5 minutes, by simple manual shaking of the dissolving container.

10 The main obstacle in the formulation of such a product is the low water solubilization rate of almost all the most-commonly used surfactants when they are in a highly concentrated form, which is, obviously, the only possible form that can be used for the product according to this invention.

The following Table 1 evidences the above statements by reporting the solution times of some presently-marketed surfactants (the indicated times are those required to obtain the solubilization in water, at room temperature, by simple manual shaking, for preparing final solutions at 14% active matter
15 concentration).

TABLE 1

20	Product	content of active agent expressed in % by weight	Solution time (minutes)
25	LAS Na,	with 60% of active agent	more than 30
	LAS Tea,	with 60% of active agent	more than 30
	LAS K/Tea,	with 60% of active agent	about 10
30	SAS Na,	with 60% of active agent	more than 30
	SAS Na,	with 93% of active agent	more than 360
	AE3S Na,	with 70% of active agent	more than 30
35			

Even by mixing some of the above mentioned surfactants with each other, no successful results are obtained; on the contrary, in certain cases, the solution time increases with respect to that of the individual
40 components, as reported in Table 2.

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TABLE 2

5	Product (mixtures 1:1)	Solution time (minutes)
10	LAS Na 60 -AE3S Na 70	about 60
	SAS Na 60 -LAS Na 60	more than 90
	SAS Na 60 -AE3S Na 70	about 60
15	SAS Na 93 -AE3S Na 70	more than 360
	LAS K/TEA 60 -AE3S Na 70	more than 30
	LAS TEA 60 -AE3S Na 70	more than 60

20

Surprisingly, it has been discovered according to the present invention, that by mixing two or more anionic surfactants, chosen from the groups of linear alkylbenzensulfonates, alkanolamine salts, alkyletoxysulfates with low ethoxylation degree sodium salts, alkansulfonates sodium salts, with one or more nonionic surfactants, chosen from the groups of ethoxylated alcohols or alkylphenols and alkanolamides, in the presence of suitable amounts of a solvent mixture composed of a lower alcohol and water, the desired result is acquired of a quick and easy solubilization in water, in times lower than 5 minutes, by simple dilution with minimum shaking.

25

It has also been discovered that, in order to achieve the above surprising results of obtaining a composition having a very high concentration of active matter, in the order of 60-85%, combined with a quick solubility in water, in times ranging from 3 to 5 minutes, by simply manual shaking of the diluting container, it is essential that one of anionic surfactants be formed by an alkylbenzensulfonate triethanolamine salt, and it is further essential that the above alkylbenzenesulfonate triethanolamine salt be formed during the preparation step of the detergent composition by direct neutralization of the corresponding sulphonic acid with the organic amine, added in stoichiometric ratio, in the presence of the other components of the composition, i.e. the nonionic surfactant(s) and the solvent (water + alcohol).

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Therefore, the object of this invention is to provide a complete detergent, characterized by the following features:

1) high concentration of active matter, above 60%, preferably from 65 to 85%, such as to allow the preparation of liquid detergents, ready for use, from relatively small volumes of product, by dilution with water, to final concentrations in the range from 10 to 30% of total active matter.

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2) A pH of the diluted detergent comprised between 6.5 and 7.5.

3) Quick solubilization of the product in water, at room temperature, by simple mixing and minimum stirring, within times of the order of 3-5 minutes.

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Among the anionic surfactants the straight-chain alkylbenzensulfonates triethanolamine salts, the sodium alkansulfonates with C13-C18 chain length and the sodium alkyletoxy sulfates, containing from 2 to 3 moles of ethylene oxide, with an alkyl chain length in the range C12-C15, of the kind derived from coconut alcohols, commercial C9-C15 or C12-C18 cuts, sulfated, sodium salts, condensed with 3 moles of ethylene oxide, have proved to be suitable.

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As non-ionic surfactants, the invention advantageously uses ethoxylated alkylphenol with from 6 to 9 moles of ethylene oxide, of the kind of the nonylphenol condensed with 9 moles of ethylene oxide; ethoxylated linear alcohols (type C9-C15) with 7 to 15 moles of ethylene oxide, of the kind of the synthetic oxo-alcohols, C9-C15 cut, condensed with 7 moles of ethylene oxide; alkanolamides of fatty acids (coconut type) of the kind of the coconut diethanolamide; or binary or ternary mixtures of the above mentioned surfactants.

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As organic solvents, lower alcohols with a number of carbon atoms from 1 to 3, of the kind of the ethanol; low molecular weight ketones such as acetone and methylethylketone, and some glycol ethers such as monomethyl- and monoethyl-glycol ethers may be used.

Hereinafter some examples of practical application of the present invention are reported, from the formulation of products which comply with the usual average level of performance which is aspected by the consumer and afforded by the commercial formulations presently on the market.

They have simply the aim of explaining more clearly the concept and possibilities of our invention,
5 without constituting in any case, limitations to its practical development.

Examples 1-2

10 Products for dishwashing were prepared by mixing together, at room temperature, the various components as indicated below (in parts per cent):

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	Component	Form. 1	Form. 2
20	Alkylbenzenesulphonic acid 95%	38.0	33.10
	Triethanolamine	16.7	14.56
25	Empimin KSN 70Z*	25.5	35.30
	Empilan NP 9**	5.0	5.00
	Ethanol	14.0	12.04
30	Ethylenglicol	0.8	--

35

The so prepared products had the following composition and physical data:

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5	Description	Product	Product
		1	2
	Active matter:		
	LAS Tea %	52.80	46.01
10	AE3S Na %	17.85	24.71
	Nonionic surfactant	5.00	5.00
	Total surfactants %	75.65	75.72
15	LAS Tea/ AE3S Na ratio	3.54/1	1.86/1
	Physical state	liquid	liquid
20	Solution times (minutes)	3	3
	Stability (at -5°C)	stable	stable

The practical washing performance of formulations 1 and 2 was evaluated on the same products, diluted to 14% active matter concentration, by direct comparison with a reference formulation, representative of a typical product in the dishwashing field on the home market, having the composition:

Alkylbenzensulphonate, Na salt (as 100%) 11.2%
 AE3S, Na salt (Empimin KSN 70-Z) as 100% 2.8%
 Minor components and water 86.0%

The washing tests were run on artificially soiled plates, where each plate was soiled with 3 g of a standard natural soil composed of the main ingredients of a typical Italian meal such as ragout (made up with beef meal, oil, butter, tomato, onion, cheese), under the following conditions:

Bath volume 1.4
 Water hardness 25°F
 Temperature 50°C
 Detergent conc. 1 g/l

The washing results, expressed as number of plates washed until the washing solution surface is only half covered with foam, were as follows:

Reference formulation 15.3 (average of six tests)
 Form. 1 14.5 (average of six tests)
 Form. 2 14.8 (average of six tests)

Examples 3-5

Other formulations for dishwashing are reported with the relative composition, physical and performance data, expressed in the same way and with the same meaning as in the preceding examples 1 and 2.

5

	Formul. (parts per cent)	3	4	5
10	Alkylbenzensulfonic acid 95%	28.0500	28.0500	28.0500
	Triethanolamine	14.5900	14.5900	14.5900
15	Empimin KSN 70-Z	29.9500	29.9500	29.9500
	Empilan NP 9	9.1200	4.5600	--
	Coconut diethanolamide	--	4.5600	9.1200
20	Ethanol	14.0000	14.0000	14.0000
	Ethyleneglycol	3.3274	3.3274	3.3274
	Perfume & dyes	0.9626	0.9626	0.9626
25	Active matter			
	LAS Tea %	38.99	38.99	38.99
	AE3S Na %	20.97	20.97	20.97
30	Nonionic %	9.12	9.12	9.12
	Total surfactants %	69.08	69.08	69.08
	LAS Tea/ AE3S ratio	1.86/1	1.86/1	1.86/1
35	Total anionic/non ionic ratio	6.6 /1	6.6 /1	6.6 /1
	pH (on product t.q.)	6.5	6.85	7.10
40	pH (on product diluted to			
	14% act. matter)	7.0	7.15	7.30
	Viscosity (20°C) cP	330	390	250
45	Solution times (20°C) minutes	3	3	2.5
	Stability (-5°C)	stable	stable	stable
	Number of plates washed, at			
50	half residual foam (average			
	out of 6 tests; reference			
	formul = 14.8)	15	15.7	16.7
55				

Example 6

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Parts per cent

10	Alkylbenzensulfonic acid 95%	23.3700
	Triethanolamine	12.1600
	Empimin KSN 70-Z	24.9800
15	Hostapur* SAS 93%	10.7500
	Empilan NP 9	4.5600
	Coconut diethanolamide	4.5600
20	Ethanol	14.0000
	Water	4.6574
25	Perfume & dyes	0.9626
	<u>*Alkan (C13-C18) sulfonate, Na salt, Hoechst trade mark</u>	
	Total surfactants %	69.08
30	LAS Tea %	32.47
	AE S Na %	17.49
	SAS %	10.00
35	Nonionic %	9.12
	<u>Total anionic/nonionic ratio</u>	<u>6.6/1</u>
	pH (on the product t.q.)	6.8
40	pH (on the product diluted to 14%	
	active matter)	7.4
	Viscosity (20°C) cP	470
45	Solution time (20°C) minutes	3.5
	Stability (-5°C)	stable
50	Number of plates washed, at half residual	
	foam (average of 6 tests; reference	
	formul = 14.8)	15.2

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Example 7

	<u>Parts per cent</u>
5	
	Alkylbenzensulfonic acid 95%
	23.95
10	Triethanolamine
	12.10
	Empimin KSN 70-Z
	15.84
	Empilan NP 9
	20.00
15	Coconut diethanolamide
	20.00
	Ethanol
	7.15
	Perfume & dyes
	0.96
20	
	<hr/>
	Total surfactants % (active matter)
	84.4
	LAS Tea %
	33.28
25	AE3S Na %
	11.09
	Nonionic %
	40.0
	LAS Tea/AE3S Na ratio
	3/1
30	<u>Total anionic/nonionic ratio</u>
	<u>1.1/1</u>
	pH (on the product t.q.)
	7.7
35	Viscosity (20°C) cP
	835
	Solution time (20°C) minutes
	4
	Stability (-5°C)
	stable
40	Number of plates washed, at half residual
	foam (average of 6 tests; reference
	formul = 14.6)
	15.5

Claims

- 50 1. A concentrated detergent composition in the form of a viscous liquid, quickly soluble in water at room temperature with minimum manual shaking, suitable for the prompt preparation within a time of from 3 to 5 minutes of diluted liquid detergents ready for use, comprising:
- a) one or more nonionic surfactants, selected from the groups of:
- 55 -ethoxylated alcohols, with alkyl chain lenght in the range C9-C18, mainly C12-C14, containing from 6 to 15 moles of ethylene oxide.
- ethoxylated alkylphenols, with alkyl chain length in the range C8-C9, containing from 6 to 15 moles of

ethylene oxide.

-mono and di ethanolamides, of fatty acids with from 10 to 16 carbon atoms.

for a total concentration of nonionic surfactants in the range 5-:-40%.

b) an organic solvent selected from the groups of lower alcohols (C2-:-C4 alkyl chain, linear or branched), glycols (C2-:-C4 alkyl chain), glycols C2-:-C4) mono and di-methyl (or ethyl) ethers, ketones with total carbon atoms numbers from 3 to 5, or mixtures thereof, at a concentration from 5 to 20%.

c) water, at concentration from 0 to 15%.

d) minor components with specific functions, such as perfumes, dyes, and the like, which do not impair the characteristics thereof.

e) -two or more anionic surfactants, one of which is a triethanolamine salt of a linear alkylbenzenesulfonate (C10-C14 alkyl chain length), which is formed during the preparation of the composition from the corresponding alkylbenzenesulfonic acid by direct neutralization with the triethanolamine, added stoichiometric ratio, in the presence of the said nonionic surfactants and of the said organic solvent; said second and further anionic surfactant being selected from the group of:

-linear alkylbenzenesulfonates (C10-C14 alkyl chain length) alkanolamine salts.

-alkyl etoxy sulfates (AExSNa), with alkyl chain length in the range C9-C18, with ethoxylation degree from 1 to 3, sodium salts.

-alkansulfonates, with alkyl chain length in the range C13-C18, sodium salts.

for a total concentration of anionic surfactants in the range 30 -:-60%

The total active matter concentration of (a + e) being in the range from 60-:-85% with a ratio anionic:nonionic surfactants ranging from 10:1 to 1:1;

The above compositions being such as to give final diluted detergents, at concentrations comprised in the range 10-:-30% active matter, preferably around 15%, with a pH comprised in the range from 6.5 to 7.5.

2. A composition according to claim 1, in which the alkylethoxysulfate derives from coconut alcohols, commercial C9-C15 or C12-C18 cuts, sulfated sodium salts, condensed with 3 moles of ethylene oxide.

3. A composition according to claim 1, in which the ratio LAS Tea/AE3S Na is in range from 9:1 to 1:1.

4. A composition according to claim 1, in which as nonionic surfactant, nonylphenol condensed with 9 moles of ethylene oxide is used.

5. A composition according to claim 1 in which, as nonionic surfactant, a coconut diethanolamide is used.

6. A composition according to claim 1 in which, as nonionic surfactants, synthetic oxo-alcohols, C9-C15 cut, condensed with 7 moles of ethylene oxide, is used.

7. A composition according to claim 1 in which binary or ternary mixtures, in any ratio, of the mentioned nonionic surfactants, are used.

8. A composition according to claim 1, in which the organic solvent is ethanol.