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CA 2230229 C 2008/01/08

(11)(21) 2 230 229

(12) BREVET CANADIEN
CANADIAN PATENT

(13) C

(86) Date de dépôt PCT/PCT Filing Date: 1996/08/20
(87) Date publication PCT/PCT Publication Date: 1997/03/13
(45) Date de délivrance/Issue Date: 2008/01/08
(85) Entrée phase nationale/National Entry: 1998/02/24
(86) N° demande PCT/PCT Application No.: EP 1996/003659
(87) N° publication PCT/PCT Publication No.: 1997/009405
(30) Priorité/Priority: 1995/09/05 (EP95202403.2)

(51) Cl.Int./Int.Cl. *C11D 3/12* (2006.01), *C11D 17/00* (2006.01), *C11D 3/37* (2006.01), *C11D 3/386* (2006.01)

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(54) Titre : COMPOSITION DETERGENTE LIQUIDE CONTENANT DE LA SILICE HYDROPHILE DISPERSEE
(54) Title: LIQUID DETERGENT COMPOSITION COMPRISING DISPERSED HYDROPHILIC SILICA

(57) Abrégé/Abstract:
Liquid detergent composition comprising from 0 to 20 % by weight of water with dispersed particles wherein the solid phase comprises a hydrophilic silica material, a method for including enzymes and a method for preparing such liquids.

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 :
C11D 3/12, 3/37, 3/386

A3

(11) International Publication Number: **WO 97/09405**

(43) International Publication Date: 13 March 1997 (13.03.97)

(21) International Application Number: PCT/EP96/03659

(22) International Filing Date: 20 August 1996 (20.08.96)

(30) Priority Data:
95202403.2 5 September 1995 (05.09.95) EP
(34) Countries for which the regional or international application was filed: NL et al.

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(81) Designated States: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).

Published
With international search report.
Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(88) Date of publication of the international search report:
9 May 1997 (09.05.97)

(54) Title: LIQUID DETERGENT COMPOSITION COMPRISING DISPERSED HYDROPHILIC SILICA

(57) Abstract

Liquid detergent composition comprising from 0 to 20 % by weight of water with dispersed particles wherein the solid phase comprises a hydrophilic silica material, a method for including enzymes and a method for preparing such liquids.

LIQUID DETERGENT COMPOSITION COMPRISING DISPERSED HYDROPHILIC SILICA

Technical Field

The present invention relates to low or no water liquid detergent compositions comprising solid particles, and to a method of preparing such particles and such liquids.

Background & Prior Art

Liquid detergent compositions with low water levels are known in the art and have been described in quite a number of patent publications, e.g. in US-A-4,316,812, US-A-4,874,537 and EP-A-0,484,095 and provide a way of concentrating liquid detergents without giving in on washing performance.

Liquid detergent compositions with low or no water levels may comprise a liquid phase and a dispersion of solid detergent components and adjuncts depending on the purpose of use, primarily surfactants and builders.

The liquid phase often comprises a nonionic surfactant as major component, which apart from acting as carrier liquid for the detergent components, usually and preferably also has detergent-active properties, thereby preferably acting wholly or in part as the surfactant ingredient. The liquid phase may comprise a nonionic surfactant and/or surfactant mixtures, such as mixtures of C13-C15 alcohols with an average of 3 ethoxy groups and C13-C15 alcohols with an average of 7 ethoxy groups and liquid phases as described in GB-A-1,462,134, WO 91/12313, WO 91/14765 and EP-A-0,510,762.

The solid phase may for example comprises of builders, bleach, abrasives, dispersants, enzymes, solid surfactant material and/or minor ingredients such as fluorescers. It has been found very difficult to stably suspend solid particles in liquid compositions and various solutions have

been proposed in the art.

Liquids with low or no water content comprising dispersed solid particles of the prior art generally contain high levels of solid material. Levels of 25% by weight or higher are generally used in order to arrive at stable dispersions of the solid particles in the liquid phase or to achieve the desired performance. High levels of solid particles may however lead to liquids with higher viscosity and/or poor dispersibility.

Various documents in the art have proposed to use silica material in liquid detergent compositions. GB 1,205,711 discloses non-aqueous liquids comprising hydrophilic silica and solid dispersed silica.

We have found that the liquids of the prior art may show setting, sedimentation, clear layer separation and/or poor dispersability.

Enzymatic liquid compositions with low or no water levels may be prepared by adding enzyme slurries to the liquid. These slurries require extra processing for preparation and consequently are more expensive. They are non-aqueous and viscous which means that they are more difficult to handle. Further, we have found that preparations with low water levels which are made with enzyme slurries may suffer from instability.

Surprisingly, we have now found that one or more of these disadvantages of liquids with low or no water levels can be overcome.

We have found that low solid levels can be stably incorporated in low or no water liquids if a hydrophilic silica is included.

Further, we have found that inclusion of silicone material improves the properties of low or no water liquids comprising solid particles.

Further, we have found that fluorescent agent and/or anti-redeposition agent can be incorporated in low or no water liquids with hydrophilic silica.

We have further found that liquids comprising 0 to 20% by weight of water and enzymes can be prepared with good properties if the liquid comprises silicone and/or hydrophilic silica.

We have further found that structured liquids with low water content with suspended enzyme particles can be prepared by precipitating the enzyme from an aqueous solution in the product. This is in particular surprising, as the skilled man would not be inclined to add water to low water containing liquids. Instead he would try to keep all ingredients and machinery as dry as possible.

We have further found that liquids comprising enzymes can be prepared by mixing the enzyme with a liquid composition which comprises silica, from 0 to 20% by weight of water and further preferably comprises silicone.

Statement of Invention

According to the present invention there is provided a liquid detergent composition comprising water, surfactant material, silicone material and 0.05 to 20% by weight of a dispersed solid phase comprising hydrophilic silica material and enzyme material, characterized in that the composition comprises less than 12% by weight of the water, at least 70% by weight of the surfactant material and 1-5% by weight of the enzyme material.

The composition may further comprise a fluorescent agent at a level of from 0.01 to 10% by weight.

The composition may further comprise an anti-redeposition agent at a level from 0.1 to 5% by weight.

The composition may comprise electrolyte material at a concentration of lower than 1% by weight.

The composition may comprise at least 0.5% by weight of water.

The invention also provides a process for preparing a liquid detergent composition as defined above, wherein said process comprises the step of adding the enzyme material in the form of an aqueous solution to a liquid composition which comprises silicone material, hydrophilic silica, surfactant material and water.

Detailed Description of the Invention

WO 94/29427 discloses liquids with high viscosity silicone droplets which may contain silica particles, and wherein the composition may further comprise hydrophobic silica particles.

WO 94/23009 discloses liquids with hydrophobic silica to which silicone oil may be added.

CA 2,066,008 discloses liquids with droplets of silicone oil which droplets may comprise silica, e.g. DB100 and Thompson-Hayward AF-IND-100.

EP 573,699 discloses silica particles (into which nonionic surfactant is absorbed) which are dispersed in silicone oil to produce antifoam particles. These particles may be included in liquids. The surfactant inside the silica prevents the silicone/silica droplets from co-agulating during storage.

EP 583,512 discloses the use of particles of silica with silicone, to protect sensitive ingredients, such as enzymes, in liquids.

GB 2,186,884 discloses particles (or droplets) of hydrophobic silica and silicone for use as enzyme stabilisers.

GB 1,205,711 discloses liquids with hydrophilic silica and enzymes.

We have found that hydrophilic silica allows for formulation of liquids comprising from 0 to 20% by weight of water and low levels of dispersed solid phase. The liquids are stable and of low viscosity with no or low setting and low or no clear layer separation, whilst they have a good dispersibility.

We have further found that silicone inclusion in liquids with hydrophilic silica and low or no water level, enables stable suspending of solid particles, whilst the resulting liquid has low viscosity, low or no setting, low or no

clear layer separation and good dispersibility.

We have further found that liquids with fluorescent agent and/or anti-redeposition agent can be stable formulated with hydrophilic silica and low or now water. Preferably, no electrolyte is present and preferably the liquid contains some water.

Hydrophilic silica material

The present invention requires the presence of hydrophilic silica, preferably present in dispersed form in the continuous phase of the liquid and forming a network which is able to disperse solid particles. Surprisingly, we have found that liquids of the invention with low solid levels, silicones, fluorescer and/or anti-redeposition agent do not suffer from the bad properties of liquids of the art comprising hydrophilic silica.

Hydrophilic silica material selected from amorphous silica, precipitated, fumed silica, gel-formation formed silica and mixtures thereof. An example of such a silica material is Aerosil 200™. Mixtures of hydrophilic and hydrophobic silica (e.g. Sypernat) may also be used, e.g. on a 50:50 basis.

Preferably, the silica level is higher than 0.5%, more preferably higher than 1.0%, most preferably higher than 2% by weight of the composition. Preferably the level is lower than 8%, more preferably lower than 6%, most preferably lower than 4% by weight of the composition.

Silicone material

WO 93/24603, WO 94/01525 and WO 94/01524 disclose non-aqueous liquids with DB100™, which is a commercially available silicone polymer material. EP-A-515,418 and EP-A-515,435 disclose the use of silicone material in non-aqueous liquids.

Preferably, the silicone material is selected from polydialkylsiloxanes, wherein the alkyl may e.g. be C1-C6 and is preferably a methyl group. Polydialkylsiloxanes may end blocking units, e.g. of a trimethylsilyl unit. An example is DB100, ex Dow. Silicone may be added to the composition in the form of a mixture with silica material.

Preferably, the silicone material is present at levels of from 0.01 to 1%, more preferably 0.05 to 0.5% by weight of the composition.

We have found that the physical properties of the liquids according to the invention may be improved by incorporation of the silicone material.

Water

Liquids of the invention may not contain water. However, preferably, the water content of liquid detergent products of the invention is from 0.05% to 20% by weight of the composition in order to provide high stability. Preferably, the water level is less than 15% by weight, more preferably less than 12% by weight, most preferably less than 10% and preferably more than 2%, 0%, more preferably more than 5% by weight of the composition. This is in particular surprising, as the skilled man would not be inclined to add water to low water containing liquids. Instead he would try to keep all ingredients and machinery as dry as possible.

Enzymes

As indicated aboves, we have found that use of aqueous enzyme preparations leads to stable enzyme liquids, e.g. as compared to use of enzyme slurries which may e.g. contain salt crystals.

Preferably, compositions according to the invention contain enzyme material. Enzymes may be selected from lipases,

amylases, proteases (10-1,000 GU/mg), cellulases and mixtures thereof. Preferably, enzyme levels are from 0.1-20%, more preferably from 1 to 5% by weight of the composition. Enzymes often contain propyleneglycol/water mixtures or the like to stabilise them. Preferably, the enzyme solution contains no more than 10% by weight of water-soluble electrolyte material.

Introduction of low levels of water in the liquids of the invention has been found beneficial in some instances to the stability of the liquid, whereas it has surprisingly been found not to negatively influence the viscosity of the composition nor the stability of the enzymes.

Liquid Phase

The liquid phase of the detergent composition of the present invention may comprise water, surfactant material and solvent material.

Liquids of the invention often and preferably comprise a liquid nonionic surfactant as major component and normally consist of a water-solubilising polyalkoxylene or a mono- or di-alkanolamide group in chemical combination with an organic hydrophobic group derived from, for example, fatty alcohols with from 9 to 15 carbon atoms (optionally branched, e.g. methyl branched), alkylphenols (preferably from 12 to 20 carbon atoms) in which the alkyl group contains from about 6 to about 12 carbon atoms, dialkylphenols in which each alkyl group contains from 6 to 2 carbon atoms, primary, secondary or tertiary aliphatic alcohols (or alkyl-capped derivatives thereof), monocarboxylic acids having from 10 to about 24 carbon atoms in the alkyl group and polyoxypropylenes.

C10-20 fatty acid mono- and dialkanolamides wherein the alkyloyl group has from 1 to 3 carbon atoms are also

common.

In polyalkoxylene containing surfactants, the polyalkoxylene moiety usually consists of an average of from 2 to 20 groups of ethylene oxide, propylene oxide groups or mixtures thereof. The latter class includes those described in European Patent Specification EP-A-0,225,654, especially for use as all or part of the liquid phase.

Especially preferred are those ethoxylated nonionics which are condensation products of fatty alcohols with from 9 to 15 carbon atoms condensed with 3 to 7 moles of ethylene oxide. Examples of those are the condensation products of C11-13 alcohols with 3 or 7 moles of ethylene oxide. These may be used as the sole nonionic surfactant or in combination with those described in EP-A-0,225,654.

Another class of suitable nonionics include the alkyl saccharides (polyglycosides/oligosaccharides) and, in particular those described in patent specifications US-A-3,640,998; US-A-3,346,558; US-A-4,223,129; EP-A-0,092,355; EP-A-0,099,183; EP-A-0,070,074; EP-A-0,070,075; EP-A-0,070,075; EP-A-0,070,076; EP-A-0,070,077; EP-A-0,075,994; EP-A-0,075,995 and EP-A-0,075,996.

A typical blend of surfactants includes a nonionic and/or non-alkoxylated anionic and/or alkoxylated anionic surfactant. Mixtures of different nonionic detergent surfactants may be used and also mixtures with other detergent surfactants such as anionic, cationic or ampholytic surfactants and soaps may also be used. Surfactants are described in "Surface Active Agents" Vol I, by Schwartz & Perry, Interscience 1949 and "Surface Active Agents" Vol II by Schwartz, Perry & Berch (Interscience 1958), in the current edition of "McCutcheon's Emulsifiers & Detergents" published by the McCutcheon division of

Manufacturing Confectioners Company or in "Tensid-Taschenbuch", H Stache, 2nd Edn., Carl Hanser Verlag, Munchen & Wien.

Preferably, the compositions of the invention comprise from 50 to 99% by weight of surfactant material, preferably the level is at most 97%, more preferably at most 95%, most preferably at most 94% and preferably, at least 70%, more preferably at least 80% by weight.

Solvent material may be included in compositions according to the present invention and in particular esters of C1-6 carboxylic acid and/or poly-carboxylic acid and an alcohol selected from dihydric alcohols, ether alcohols and sterically hindered alcohols. Preferably, the ester is present in amounts of from 0.01 to 50% by weight of the composition. A preferred ester material is propylene glycol di-acetate.

Other liquid material which may be present in the liquid phase include liquid bleach precursors such as for example glyceroltriacetate and solvent material like ethanol and dodecanol.

For the purpose of the invention, liquid detergent compositions are defined as being in a liquid to paste form, preferably in a liquid form. The viscosity of the compositions is preferably at most 2,500 mPa.s, more preferably at most 1,500 mPa.s, most preferably at most 1,000 mPa.s at 21s-1 at 25°C.

Preferably, liquids of the invention have a physical stability of no or minimal phase separation after 2 months at 37°C. More preferably, the phase separation is less than 2mm per month at 37°C.

Solid Phase

Preferably, compositions according to the invention comprise from 0.1 to 20% by weight of a solid phase. Preferably the level of the solid phase is at least 0.2%, more preferably at least 0.5% and, preferably, at most 15%, more preferably less than 10%, most preferably at most 9%, in particular preferred at most 5% by weight of the composition. We have found that these low levels of solid particles are beneficial not only to the viscosity of the liquid, but also to the physical stability, whilst it would be expected that higher of dispersed solid particles would be required.

Other Ingredients

Compositions of the invention may comprise electrolyte material. We have however found that electrolyte may affect the properties of the compositions of the invention, in particular upon storage. Therefore, compositions of the invention preferably comprise electrolyte at a concentration of lower than 1% by weight of the composition.

More specifically, we have found that water-soluble electrolyte may affect the composition properties. Examples of such electrolytes are chloride, sulphate, carbonate and hydroxide. Consequently, the composition preferably comprise electrolyte, more particular soluble electrolyte at a concentration of lower than 1% by weight, more preferably lower than 0.3% most preferably lower than 0.2% by weight of the composition.

Furthermore, we have found that the preferred weight ratio of water:electrolyte in the compositions of the invention is more than 1:0.03, more preferably more than 1:0.02 and preferably 1:0 or less.

Other ingredients comprise those remaining ingredients which may be used in liquid detergent products, such as fabric conditioning agents, abrasive material, bleaches, builders, enzymes, enzyme stabilising agents (if necessary), fluorescers, perfumes (including deoperfumes), micro-biocides, soil-suspending agents, anti-redeposition agent, corrosion inhibitors, and lather depressants.

Enzymes which can be used in liquids according to the present invention include proteolytic enzymes (protease), amylolytic enzymes (amylase), lipolytic enzymes (lipases) and cellulolytic enzymes (cellulase). Various types of proteolytic enzymes and amylolytic enzymes are known in the art and are commercially available. They may be incorporated as "prills", "marumes" or suspensions e.g. The preferred level of enzyme materials is from 0.01 to 5% by weight of the composition.

Enzyme stability is preferably higher than 50% after 3 months storage at 37°C, more preferably higher than 80%, most preferably higher than 90%.

The total amount of the fluorescent agent or agents used in a detergent composition is preferably from 0.1 to 10%, more preferably 0.02 to 2% by weight.

When it is desired to include anti-redeposition agents in the liquid detergent products, the amount thereof is normally from about 0.1% to about 5% by weight, preferably from about 0.2% to about 2.5% by weight of the total liquid composition. Preferred anti-redeposition agents include carboxy derivatives of sugars and celluloses, e.g. sodium carboxyalkyl (e.g. methyl) cellulose, anionic poly-electrolytes, especially polymeric aliphatic carboxylates, or organic phosphonates.

Processing

As indicated, the present invention further relates to a method of preparing liquids comprising 0 to 20% by weight of water and enzymes, by adding enzyme to the liquid. Preferably, the liquid further comprises silicone and/or hydrophilic silica. Preferably, the enzyme is added in the form of an aqueous solution.

A further embodiment of the present invention is that the enzyme can be included in a structured liquid composition comprising 0 to 20% by weight of water by precipitation the enzyme from an aqueous solution in the composition.

A further embodiment is a method of preparing liquids comprising enzymes by mixing the enzyme with a liquid composition comprising silica and 0 to 20% by weight of water and preferably silicones.

The enzymes are added after silica and silicones dispersal is complete and at temperatures of 30°C or lower.

Preferably, the enzyme is in-situ precipitated. We have found that this leads to small enzyme particles that can stably be incorporated in the hydrophilic silica structure in the liquids with water levels of from 0 to 20% by weight.

The following examples are intended to further illustrate and describe the invention and are not intended to limit the invention in any way.

EXAMPLE

Example 1

The following liquids were prepared:

	Composition	
	A	1
Ingredients	(% by weight)	
Nonionic 1 1)	57.2	56.7
Nonionic 2 2)	35.9	35.4
Water	1.9	1.8
Aerosil 200	2	3
Enzyme 3)	3	-
Enzyme 4)	-	3
Silicone 5)	-	0.1
Total water	8%	10%
Total electrolyte	1.8%	0.06%
Viscosity 6)	500	250

1) Marlipal™ 013-89 (90%)

2) Marlipal 013-30 (100%)

3) Savinase™ 16.0SL (enzyme slurry)

4) Savinase 16.0LDX (aqueous/propyleneglycol enzyme preparation)

5) DB100 silicones

6) As measured with a Haake MVIIST™ after 5 minutes.

Composition 1 was prepared by dispersing the silica and silicones in the mixture of nonionic surfactants and water. Thereafter, the aqueous enzyme solution was added and the enzyme was in-situ precipitated at a temperature under 30°C.

Composition A is instable, whereas composition 1 according to the present invention is stable, shows no setting, no sediment, no clear layer separation and good dispersability.

Example 2

The following liquid was prepared by adding the ingredients in the order listed:

		Composition				
		1	2	3	4	B
Ingredients		(% by weight)				
Nonionic 1	1)	56.7	56.7	56.7	56.7	56.7
Nonionic 2	2)	35.4	35.4	35.4	35.4	35.4
Water		1.8	1.8	1.8	1.8	1.8
Aerosil 200		3	3	3	3	3
Silicone	3)	0.1	0.1	0.1	0.1	0.1
Enzyme	4)	3	3	3	3	3
Fluorescer	5)	1	1	1	-	-
Fluorescer	6)	-	-	-	1	-
SCMC	7)	1	0.5	-	-	-
SCMC	8)	-	-	-	-	1
Total water		10	10	10	10	10
Total electrolyte		0.08	0.07	0.06	0.06	0.31
Viscosity	9)	<.....thin liquids.....>				
Stability		OK	OK	OK	OK	unstable

1) Marlipal 013-89 (90%)

2) Marlipal 013-30 (100%)

3) DB100 silicones

4) Savinase 16.0LDX (aqueous enzyme solution)

5) CBS/X™ fluorescer

6) Blancophor™ MBBH766

7) Blanose™ 7M1, ex Aqualon (Hercules)

8) Gabrosa™ ex AKZO Nobel

9) Viscosity between 100-300 mPa.s at 21s-2 at 25°C

Compositions 1, 2, 3 and 4 showed good stability upon storage, whereas composition B containing higher levels of electrolyte, showed instability after 2 days of storage.

Example 3

The following liquid was prepared by adding the ingredients in the order listed:

		Composition			
		1	2	3	4
Ingredients		(% by weight)			
Nonionic 1	1)	56.7	56.7	56.7	56.7
Nonionic 2	2)	35.4	35.4	35.4	35.4
Water		1.8	1.8	1.8	1.8
Aerosil 200		3	3	3	3
Silicone	3)	0.1	0.1	0.1	0.1
Enzyme	4)	3	3	3	3
SCMC	5)	0.5	2.0	3.0	4.0
Total water		10	10	10	10
Total electrolyte		0.07	0.10	0.12	0.14
Viscosity 6)		<.....thin liquids.....>			

1) Marlipal 013-89 (90%)
2) Marlipal 013-30 (100%)
3) DB100 silicones
4) Savinase 16.0LDX (aqueous enzyme solution)
5) Blanose 7M1, ex Aqualon (Hercules)
6) Viscosity between 100-350 mPa.s at 21s-2 at 25°C.

The four compositions showed good stability after 3 weeks storage at 37°C.

CLAIMS

1. Liquid detergent composition comprising water, surfactant material, silicone material and 0.05 to 20% by weight of a dispersed solid phase comprising hydrophilic silica material and enzyme material, characterized in that the composition comprises less than 12% by weight of the water, at least 70% by weight of the surfactant material and 1-5% by weight of the enzyme material.

2. Composition according to claim 1, wherein the composition further comprises a fluorescent agent at a level of from 0.01 to 10% by weight.

3. Composition according to claim 1 or 2, wherein the composition further comprises an anti-redeposition agent at a level of from 0.1 to 5% by weight.

4. Composition according to any one of claims 1 to 3, wherein the composition comprises electrolyte material at a concentration of lower than 1% by weight.

5. Composition according to any one of claims 1 to 4, wherein the composition comprises at least 0.5% by weight of water.

6. Process for preparing a liquid detergent composition according to any one of claims 1 to 5, wherein said process comprises the step of adding the enzyme material in the form of an aqueous solution to a liquid composition which comprises silicone material, hydrophilic silica, surfactant material and water.