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(54) **DETERGENT COMPOSITION**

WASCHMITTELZUSAMMENSETZUNG

COMPOSITION DETERGIVE

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DescriptionTECHNICAL FIELD

[0001] The present invention relates to substantially non-aqueous liquid cleaning products, especially detergent compositions and to a method of preparing a non-aqueous liquid. Non-aqueous liquids are those containing little or no water, usually less than 5% by weight, preferably less than 3% by weight, more preferably less than 1% by weight of the composition.

PRIOR ART & BACKGROUND

[0002] Non-aqueous liquid detergent compositions 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. Non-aqueous liquids provide a way of concentrating liquid detergents without giving in on washing performance.

[0003] Non-aqueous liquid detergent compositions normally comprise a non-aqueous liquid phase having incorporated therein as dispersion, solution or combination thereof, the usual detergent components and adjuncts depending on the purpose of use, primarily surfactants and builders.

[0004] 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 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.

[0005] EP-A-510,762 discloses non-aqueous liquids comprising solid dispersed particles and a polymer material to overcome physical stability.

[0006] WO 93/24603, WO 94/01525 and WO 94/01524 disclose non-aqueous liquids with DB100® a low viscosity silicone polymer material having a viscosity of 3,000 mPa.s at 21s⁻¹ at 25°C.

[0007] EP-A-515,418 and EP-A-515,435 disclose the use of silicone material in non-aqueous liquids.

[0008] Non-aqueous liquids of the prior art comprising silicone polymer material may suffer from sedimentation, clear layer formation or poor dispersibility. Surprisingly we have found that one or more of these disadvantages can be overcome.

SUMMARY OF THE INVENTION

[0009] The invention provides a non-aqueous liquid cleaning composition, according to claim 1.

[0010] Preferably the viscosity of the droplets is of from 5,000 to 60,000 mPa.s at 21s⁻¹ at 25°C.

DETAILED DESCRIPTION OF THE INVENTION

[0011] Although not wishing to be bound by any theory, it is believed that gluing together of solid particles dispersed in a non-aqueous environment is reduced by using dispersed droplets according to the invention.

Dispersed droplets

[0012] The dispersed droplets according to the invention comprise silicone polymer and hydrophobic particles and have a viscosity of at least 5,000 mPa.s at 21s⁻¹ at 25°C, preferably at least 10,000, more preferably at least 15,000, most preferably at least 20,000. Preferably the viscosity of the dispersed droplets is at most 60,000 mPa.s at 21s⁻¹ at 25°C, preferably at most 50,000 mPa.s at 21s⁻¹ at 25°C, more preferably at most 40,000 mPa.s, most preferably at most 35,000 mPa.s.

[0013] Preferably the dispersed droplets also comprise alkoxypoly-dimethyl stabilising agents, e.g. DC-198 (ex Dow Corning), which is believed to be able to further improve the properties of the non-aqueous liquids as to sedimentation, clear layer formation and/or poor dispersibility.

[0014] The weight ratio of the silicone polymer to the hydrophobic particles is from 70:30 to 99:1, preferably from 80:20 to 98:2, more preferably from 90:10 to 95:5.

[0015] The dispersed droplets are present in amounts of from 0.1 to 5.0% by weight of the composition, preferably 0.3 to 3.0% by weight, preferably from 0.5 to 2.0% by weight.

[0016] Preferably the particle size of the droplets is from 0.5-50 µm (in terms of D(1.0), being the number mean diameter as described by M. Alderliesten, Anal. Proc. Vol. 21, May, 1984, 167-172, as can be microscopically determined using a fluorescence imagin technique; images can be digitised so that droplet areas and diameters can be

measured and counted using image analysis), preferably from 1-30µm.

[0017] Surprisingly, we have found that high viscosity droplets according to the invention can be prepared by blending a conventional low viscosity silicone polymer composition with a high viscosity fluid. Optionally, the solid content of the composition, e.g. hydrophobic particles, can be adjusted.

Silicone polymer

[0018] Preferably, the silicone polymer comprises an organosiloxane polymer, more preferably a polydimethylsiloxane.

[0019] The polymer may also consist of a mixture of polymers, e.g. a mixture of a polymer that has a high viscosity (due to branching or to a high molecular weight) per se, e.g. more than 5.000 mPa.s at 21 s⁻¹ at 25°C, and a polymer that has a low viscosity per se, e.g. say lower than 5.000 mPa.s at 21 s⁻¹ at 25°C. An example of a high viscosity polymer is polydimethylsiloxane of a weight average molecular weight of 137,000 that has a viscosity of 60,000 mPa.s at 21 s⁻¹ at 25°C. DB100® (ex Dow Corning) comprises a polydimethylsiloxane with a weight average molecular weight of 37,000. These two polymers could for example be mixed in a 50/50 w/w ratio.

Hydrophobic particles

[0020] Preferably, the hydrophobic particles are selected from hydrophobic silica, hydrophobed metal oxide (e.g. aluminium, titanium and magnesium), α-w dialkylamide alkane, polyvalent metal salt of an alkyl phosphoric acid and polyvalent metal salt of an alkyl carboxylic acid. The most preferred particle to be used in the dispersed droplets is hydrophobic silica

[0021] Preferably the particle size of the hydrophobic particles is smaller than 10µm, more preferably smaller than 1µm.

Non-aqueous liquid phase

[0022] The non-aqueous liquid phase of the compositions according to the present invention may comprise surfactant and other liquid materials, such as liquid bleach precursors solvents and deflocculant material.

[0023] The non-aqueous liquid phase will generally be present in amounts of at least 10%, preferably 20%, more preferably 30% most preferably 35% by weight of the non-aqueous liquid composition and generally at most 90%, preferably 70%, more preferably 60% and most preferably 50% by weight of the composition.

[0024] Generally, non-aqueous liquid detergent products have a free water content of less than 5% by weight, preferably less than 2% by weight, more preferably substantially nil.

Surfactants

[0025] As explained hereinbefore the liquid phase of non-aqueous liquid detergent compositions comprises a liquid nonionic surfactant as major component.

[0026] Nonionic detergent surfactants are well-known in the art. They normally consist of a water-solubilizing polyalkoxyethylene or a mono- or di-alkanolamide group in chemical combination with an organic hydrophobic group derived, for example, from alkylphenols in which the alkyl group contains from about 6 to about 12 carbon atoms, dialkylphenols in which each alkyl group contains from 6 to 12 carbon atoms, primary, secondary or tertiary aliphatic alcohols (or alkyl-capped derivatives thereof), preferably having from 8 to 20 carbon atoms, monocarboxylic acids having from 10 to about 24 carbon atoms in the alkyl group and polyoxypropylenes. Also common are fatty acid mono- and dialkanolamides in which the alkyl group of the fatty acid radical contains from 10 to about 20 carbon atoms and the alkyl group having from 1 to 3 carbon atoms. In any of the mono- and di- alkanolamide derivatives, optionally, there may be a polyoxyalkylene moiety joining the latter groups and the hydrophobic part of the molecule. In all polyalkoxyethylene containing surfactants, the polyalkoxyethylene moiety preferably consists of from 2 to 20 groups of ethylene oxide or of ethylene oxide and propylene oxide groups. Amongst the latter class, particularly preferred are those described in EP-A-225,654, especially for use as all or part of the liquid phase. Also preferred are those ethoxylated nonionics which are the condensation products of fatty alcohols with from 9 to 15 carbon atoms condensed with from 3 to 11 moles of ethylene oxide. Examples of these are the condensation products of C₁₁₋₁₃ alcohols with (say) 3 or 7 moles of ethylene oxide. These may be used as the sole nonionic surfactants or in combination with those of the described in the last-mentioned European specification, especially as all or part of the liquid phase.

[0027] Another class of suitable nonionics comprise the alkyl polysaccharides (polyglycosides/oligosaccharides) such as described in any of specifications US 3,640,998; US 3,346,558; US 4,223,129; EP-A-92,355; EP-A-99,183; EP 70,074, '75, '76, '77; EP 75,994, '95, '96.

[0028] Mixtures of different nonionic detergent surfactants may also be used. Mixtures of nonionic detergent surfactants with other detergent surfactants such as anionic, cationic or ampholytic detergent surfactants and soaps may also be used.

[0029] Preferably the level of nonionic surfactants is from 10-90% by weight of the composition, more preferably from 20-70%, most preferably from 35 to 50%.

[0030] While nonionic surfactants are quite effective at oily and greasy soil removal (e.g. sebum), particulate soils, such as clay soils and the like, may be more effectively removed by anionic surfactants. Thus within the ambit of the present invention, a very wide variation in surfactant types and levels is possible. The selection of surfactant types and their proportions will be fully within the capability of those skilled in the art.

[0031] Useful composition within the invention normally comprise blends of different surfactant types. Typical blends include those where the primary surfactants comprise a nonionic and/or non-alkoxylated anionic and/or alkoxylated anionic surfactant. Cationic, zwitterionic and amphoteric surfactants may also be present usually in minor amounts as desirable. These and other surfactants are described in "Surface Active Agents" Vol. I, by Schwartz & Perry, Interscience 1949; "Surface Active Agents" Vol. II, by Schwartz, Perry & Berch, Interscience 1958; the current editions of "McCutcheon's Emulsifiers & Detergents" published by the McCutcheon Manufacturing Confectioners Company; in "Tensid-Taschenbuch", H. Stache, 2nd Edition, Carl Hanser Verlag, Munchen & Wien, 1981; and in the various patent literature describing various types of liquid detergent compositions, which for the purpose of the invention need no further detailing.

Other liquid materials

[0032] Examples of other liquid materials which may be present in the liquid phase are liquid bleach precursors such as for example glyceroltriacetate, solvent materials for example ethanol and dodecanol and deflocculant material, as described in EP-A-266199 (Unilever), e.g. deflocculants selected from Bronsted acids and Lewis acids.

[0033] The level of liquid bleach precursors is preferably 0-20% by weight, more preferably 1-25%, most preferably 2-10%.

[0034] The level of solvents other than nonionic surfactants is preferably from 0-20%, most preferably 0-15%, more preferably 0-10% by weight.

[0035] Deflocculant material may be present, preferably at levels of from 0-15% by weight, in many cases the level is at least 0.01%, usually 0.1% or more preferred at least 1% by weight, and may be as high as 15% by weight. For most practical purposes, the amount ranges from 2-12%, preferably from 4-10% by weight, based on the final composition.

Solid particles

[0036] The compositions comprise a solid dispersed phase. The solid phase is present in amounts as low as 1% by weight, however at least 10% is preferred and more preferably 20%, most preferably 30% by weight of the composition and at most 70% and particularly preferred 60% by weight of the composition.

[0037] The solid phase may comprise one or more ingredients selected from bleach materials, solid bleach activators, builders, abrasives, fabric softening material, enzymes and minor ingredients such as fluoescers, which particles size may not necessarily be the same as that of the biopolymer material.

[0038] Usually the particle size of the solid phase defined in terms of D(3,2) will be less than 100µm, preferably not more than 30µm, more preferably up to 10µm. Usually the D(3,2) of the solid phase is more than 0.1µm, preferably at least 1µm and more preferably at least 2.5µm. For the purpose of the present invention references to the D(3,2) average particle diameter refer to the D(3,2) particle size, which is the average surface weighted, volume/weight mean diameter calculated as described by M. Alderliesten, Anal. Proc. Vol. 21, May, 1984, 167-172. The particle size can for example be determined by using a Malvern Mastersizer or a Coulter LS 130, as appropriate.

Bleaches

[0039] Bleaches include the halogen, particularly chlorine bleaches such as are provided in the form of alkalimetal hypohalites, e.g. hypochlorites. In the application of fabrics washing, the oxygen bleaches are preferred, for example in the form of an inorganic persalt, preferably with a bleach precursor or as a peroxy acid compound.

[0040] In the case of the inorganic persalt bleaches, the bleach precursor or activator makes the bleaching more effective at lower temperatures, i.e. in the range from ambient temperature to about 60°C, so that such bleach systems are commonly known as low-temperature bleach systems and are well-known in the art. The inorganic persalt such as sodium perborate, both the monohydrate and the tetrahydrate, acts to release active oxygen in solution, and the activator is usually an organic compound having one or more reactive acyl residues, which cause the formation of

peroxy acids, the latter providing for a more effective bleaching action at lower temperatures than the peroxybleach compound alone. The ratio by weight of the peroxybleach compound to the activator is from about 20:1 to about 1:1, preferably from about 10:1 to about 1.5:1. The preferred level of the peroxybleach compound in the composition is from 0-30% by weight, more preferably 2-20%, most preferably 4-15%, while the preferred level of the activator is from 0-20% by weight, more preferably 1-10%, most preferably 2-8%.

[0041] Typical examples of the suitable peroxybleach compounds are alkalimetal perborates, both tetrahydrates and monohydrates, alkali metal percarbonates, persilicates and perphosphates, of which sodium perborate and sodium percarbonate are preferred. A preferred bleach activator is TAED.

[0042] A further preferred class of bleach activators is that of hydrophobic peroxy acid bleach precursors, such as sodium nonanoyloxy benzene sulphonate and sodium -3,5,5- trimethyl hexanoyloxy benzene sulphonate. These activators are deemed to cause less (local) dye damage.

[0043] In certain cases and for particular reasons it may be desirable to also include a bleach catalyst, such as a transition metal compound or complex and the sulphonimines as described in US Patents 5,041,232 and 5,047,163, which may be used instead of or together with said bleach activators. A specifically preferred bleach catalyst for use herein is a manganese complex of formula $[\text{Mn}^{\text{IV}}_2(\mu\text{-O})_3(\text{Me-MeTACN})_2](\text{PF}_6)_2$ as described in EP-A-0,458,397 and EP-A-0,458,398. Another preferred bleach catalyst is a manganese complex as described in EP-A-549,272 (co-pending GB patent application 9127060.3). Alternatively the ligand and a manganese source can be separately added such as is described in EP-A-549,271 (co-pending GB application 9204706.7).

[0044] It is particularly preferred to include in the compositions, a stabiliser for the bleach or bleach system, for example hydroxyethylidene-1,1-diphosphonic acid, ethylene diamine tetramethylene phosphonate and diethylene triamine pentamethylene phosphonate or other appropriate organic phosphonate or salt thereof, such as the Dequest® range hereinbefore described. These stabilisers can be used in acid or salt form, such as the calcium, magnesium, zinc or aluminium salt form. The stabiliser may be present at a level of up to about 1% by weight, preferably between about 0.1 % and about 0.5% by weight.

Detergency builders

[0045] The detergency builders are those materials which counteract the effects of calcium, or other ion, water hardness, either by precipitation or by an ion sequestering effect. They comprise both inorganic and organic builders. They may also be sub-divided into the phosphorus-containing and non-phosphorus types, the latter being preferred when environmental considerations are important.

[0046] In general, the inorganic builders comprise the various phosphate-, carbonate-, silicate-, borate- and aluminosilicates-type materials, particularly the alkali-metal salt forms. Mixtures of these may also be used.

[0047] Examples of phosphorus-containing builders, when present, include the water-soluble salts, especially alkali metal pyrophosphates, orthophosphates, polyphosphates and phosphonates. Specific examples of inorganic phosphate builders include sodium and potassium triphosphates, phosphates and hexametaphosphates.

[0048] Examples of non-phosphorus-containing inorganic builders, when present, include water-soluble alkali metal carbonates, bicarbonates, borates, silicates, metasilicates, and crystalline and amorphous aluminosilicates. Specific examples include sodium carbonate (with or without calcite seeds), potassium carbonate, sodium and potassium bicarbonates, silicates such as sodiummetasilicate and zeolites.

[0049] Examples of organic builders include the alkali metal, ammonium and substituted ammonium, citrates, succinates, malonates, fatty acid sulphonates, carboxymethoxy succinates, ammonium polyacetates, carboxylates, polycarboxylates, aminopolycarboxylates, polyacetyl carboxylates and polyhydroxysulphonates. Specific examples include sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylenediaminetetraacetic acid, nitrilotriacetic acid, oxydisuccinic acid, melitic acid, benzene polycarboxylic acids and citric acid. Other examples are organic phosphonate type sequestering agents such as those sold by Monsanto under the tradename of the Dequest® range and alkanedihydroxy phosphonates.

[0050] Other suitable organic builders include the higher molecular weight polymers and co-polymers known to have builder properties, for example appropriate polyacrylic acid, polymaleic acid and polyacrylic/polymaleic acid co-polymers and their salts, such as those sold by BASF under the Sokalan® Trade Mark. Polyacrylates or their derivatives may also be useful for their anti-ashing properties.

[0051] Preferably the level of builder materials is from 5-50% by weight of the composition, more preferably 10-40%, most preferably 15-35%.

Other optional ingredients

[0052] Other ingredients comprise those remaining ingredients which may be used in liquid cleaning products, such as fabric conditioning agents, enzymes, perfumes (including deoperfumes), fluorescent agent, micro-biocides, colour-

ing agents, soil-suspending agents (anti-redeposition agent), corrosion inhibitors, enzyme stabilising agents, and lather depressants.

[0053] Amongst the fabric conditioning agents which may be used, either in fabric washing liquids or in rinse conditioners, are fabric softening materials such as fabric softening clays, quaternary ammonium salts, imidazolinium salts, fatty amines and cellulases.

[0054] 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.. Preferably enzymes are added as suspensions in a non-aqueous liquid surfactant. The preferred level of enzyme materials is from 0.01 to 5% by weight of the composition.

[0055] The total amount of the fluorescent agent or agents used in a detergent composition is generally from 0.02-2% by weight.

[0056] When it is desired to include anti-redeposition agents in the liquid cleaning 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 carboxymethyl cellulose, anionic poly-electrolytes, especially polymeric aliphatic carboxylates, or organic phosphonates.

USE

[0057] Composition in accordance with the present invention may be used for several detergency purposes, for example the cleaning of surfaces and the washing of fabrics. For the washing of fabrics, preferably an aqueous liquor containing 0.1 to 10 %, more preferably 0.2 to 2%, of the non-aqueous detergent composition of the invention is used.

PROCESSING

[0058] During manufacture of the non-aqueous liquid, it is preferred that all raw materials should be dry and (in the case of hydratable salts) in a low hydration state, e.g. anhydrous phosphate builder, sodium perborate monohydrate and dry calcite abrasive, where these are employed in the composition. In a preferred process, the dry, substantially anhydrous solids are blended with the liquid phase in a dry vessel. If deflocculant materials are used, these should preferably -at least partly- be mixed with the liquid phase, prior to the addition of the solids. In order to minimise the rate of sedimentation of the solids, this blend is passed through a grinding mill or a combination of mills, e.g. a colloid mill, a corundum disc mill, a horizontal or vertical agitated ball mill. A preferred combination of such mills is a colloid mill followed by a horizontal ball mill since these can be operated under the conditions required to provide a narrow size distribution in the final product. Of course particulate material already having the desired particle size need not be subjected to this procedure and if desired, can be incorporated during a later stage of processing.

[0059] During this milling procedure, the energy input results in a temperature rise in the product and the liberation of air entrapped in or between the particles of the solid ingredients. It is therefore highly desirable to mix any heat sensitive ingredients into the product after the milling stage and a subsequent cooling step. It may also be desirable to de-aerate the product before addition of these (usually minor) ingredients and optionally, at any other stage of the process. Typical ingredients which might be added at this stage are perfumes and enzymes, but might also include highly temperature sensitive bleach components or volatile solvent components which may be desirable in the final composition. However, it is especially preferred that volatile material be introduced after any step of de-aeration. Suitable equipment for cooling (e.g. heat exchangers) and de-aeration will be known to those skilled in the art.

[0060] It follows that all equipment used in this process should preferably be completely dry special care being taken after any cleaning operations. The same is true for subsequent storage and packing equipment.

[0061] The particles comprising carrier material and dye material associated thereto, according to the invention, can be prepared separately, as discussed above, whereafter they are dispersed in the non-aqueous phase of the non-aqueous cleaning product, e.g. by mixing using high shear or using a mill. Preferably, the D(3,2) particle size is of from 0.1 µm to 2000 µm in the non-aqueous phase.

[0062] A further embodiment of the invention provides a method of preparing a non-aqueous liquid detergent composition, according to claim 8.

[0063] Surprisingly we have found that post-dosing the silicone polymer not only make the milling step more efficient, but also improves sedimentation, lowers clear layer formation and/or poor improves dispersibility of the non-aqueous liquid.

[0064] The invention will now be illustrated with respect to the following non-limiting examples.

Example 1

[0065] The following formulation was prepared in a conventional way (including a ball-mill step) and the mixture of the silicone and hydrophobic particles was post-dosed and mixed for 10 minutes with a tip speed of 2m/s (standard to paddle arrangement):

Ingredient	% w/w
Nonionic ¹⁾	22.40
Nonionic ²⁾	27.30
LAS acid	6.00
Na Carbonate	16.40
Ca Carbonate	6.00
SCMC	1.50
Anti-ash polymer	1.50
CBS-X (fluorescer)	0.15
Silica ³⁾	4.00
Na Perborate 1H ₂ O	10.50
Enzymes	1.05
Perfume	0.50
Q2-3302 ⁴⁾	2.70

¹⁾ Vista®1012-62

²⁾ Dobanol®25-3

³⁾ Sipemat®D17 ex Degussa

⁴⁾ mixture comprising hydrophobic silica and polydimethylsiloxane ex Dow Corning (density 0.98-1.02; viscosity about 30.000mPa.s)

[0066] The non-aqueous liquid of the Example showed good properties with regard to sedimentation and dispersibility.

Example 2

[0067] The formulation of Example 1 (without enzymes and perfume) was prepared with the following changes:

- Composition A: without Q2-3302; with 2.7% by wt of DB100®⁵⁾
 Composition 1: with 2.7% Q2-3302 (post-dosed, as in Ex. 1)
 Composition 2: with 2.7% Q2-3302 (ball-milled together with composition)
 Composition 3: with 2.7% Q2-3302 and 1.2% DC198⁶⁾ (both post-dosed).
 Composition B: same as for composition A, but with thorough de-aeration after preparation.
 Composition 4: same as for composition 1, but with thorough de-aeration after preparation.

[0068] The compositions were compared in a model experiment in which clear layer formation was measured with stoppered glass 100 cm³ measuring cylinders containing 80cm³ of the product. These were suspended vertically in a glass-sided water bath and thermostatted at 37°C. Compositions A, 1, 2 and 3 were stored for 12 weeks and compositions B and 4 were stored for 4 weeks. The height in centimeters of the liquid meniscus and the clear layer separation layer was measured in duplo using a cathetometer (clear layer separation of Composition 2 was only determined once).

[0069] The results are shown in the attached figure. The composition is identified on the X-axis, the clear layer separation on the Y-axis in cm.

[0070] It is clear that compositions 1, 2, 4 and in particular 3 have a lower clear layer separation than of the comparative compositions A and B.

Example 3

[0071] DB100® (viscosity of 3000mPa.s) was blended with silicone fluid (viscosity 60000 mPa.s) using a Haake Rheocord 90 with a Rheomix 3000E head with Z blade paddles rotating at 100 rpm. Three batches were made, two consisting of a 50/50 by weight mixture of DB100® to silicone fluid and one consisting of a 43/57 by weight mixture.

⁵⁾ low viscosity droplets of silicone polymer and hydrophobic particles: viscosity of 3.000 mPa.s at 21s⁻¹ at 25°C: ex Dow Corning;

⁶⁾ alkoxypoly-dimethyl stabilising agents: ex Dow Corning.

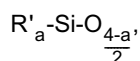
One of the 50/50 mixtures was blended for 30 minutes after addition of the fluid to the DB100®, the other for 45 minutes after addition of DB100® to the fluid. The 43/57 mixture was also blended for 45 minutes after addition of DB100® to the fluid. In all instances, addition of one ingredient to the other was accompanied by continuous mixing. Viscosity of the blends was found to be about 20,500 mPa.s at 21s⁻¹ at 25°C for the 50/50 mixtures and 25,600 mPa.s at 21s⁻¹ at 25°C for the 43/57 mixture.

[0072] This Example shows that a high viscosity silicone polymer composition can be made by blending a conventional silicone polymer with a high viscosity fluid. Optionally, the solid content of the composition, e.g. hydrophobic particles, can be adjusted.

Claims

1. Non-aqueous liquid cleaning composition comprising a non-aqueous liquid phase, from 1 to 70% by weight of solid particles and dispersed droplets comprising silicone polymer and hydrophobic particles, said droplets having a viscosity of at least 5000 mPa.s at 21s⁻¹ at 25°C, said liquid phase further comprising a liquid nonionic surfactant, with the proviso that the liquid nonionic surfactant does not consist solely of

(I) a polyorganosiloxane polyoxyalkylene copolymer, which is neither fully soluble in an aqueous surfactant solution nor fully soluble in a liquid branched polyhydrocarbyl siloxane material having a maximum of 2 mole % branching wherein at least 50% of all units present have the general formula $R_2-Si-O_{2/2}$ wherein R denotes a monovalent hydrocarbon group having up to 24 carbon atoms and wherein at least 2 units have the general structure $R-Si-O_{3/2}$ or $Si-O_{4/2}$, any other units having the general formula



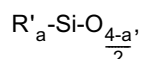
wherein $\underline{a}$ has a value of from 0 to 3 and R' denotes a group R or a hydrogen or hydroxyl group ;
or

(II) a siloxaneoxyalkylene block copolymer surfactant having a hydrophilic-lipophilic balance (HLB) of from 4 to 14.

2. Composition according to claim 1, wherein the droplets have a viscosity of from 5,000 to 60,000 mPa.s at 21s⁻¹ at 25°C.
3. Composition according to claims 1-2, wherein the hydrophobic particles are hydrophobic silica.
4. Composition according to claims 1-3, wherein the silicone polymer is polydimethylsiloxane.
5. Composition according to claims 1-4, wherein the droplets are present in an amount of from 0.1 to 5.0% by weight of the composition.
6. Composition according to claims 1-5, wherein the weight ratio of the silicone polymer to the hydrophobic particles is from 70:30 to 99:1.
7. Composition according to claims 1-6, wherein the polymer comprises a mixture of a high viscosity polydimethylsiloxane and a low viscosity polydimethylsiloxane.
8. Method of preparing a non-aqueous liquid detergent composition comprising a non-aqueous liquid phase, from 1 to 70% by weight of solid particles and dispersed droplets, said droplets comprising silicone polymer and hydrophobic particles and having a viscosity of at least 5000 mPa.s at 21 s⁻¹ at 25°C, wherein the solid particles are mixed with the non-aqueous liquid phase and optionally milled before, during or after the mixing with the non-aqueous liquid phase, whereafter the droplets are dispersed in the non-aqueous liquid phase, said liquid phase further comprising a liquid nonionic surfactant, with the proviso that the liquid nonionic surfactant does not consist solely of

(I) a polyorganosiloxane polyoxyalkylene copolymer, which is neither fully soluble in an aqueous surfactant solution nor fully soluble in a liquid branched polyhydrocarbyl siloxane material having a maximum of 2 mole % branching wherein at least 50% of all units present have the general formula $R_2-Si-O_{2/2}$ wherein R denotes

a monovalent hydrocarbon group having up to 24 carbon atoms and wherein at least 2 units have the general structure $R-Si-O_{3/2}$ or $Si-O_{4/2}$, any other units having the general formula



wherein a has a value of from 0 to 3 and R' denotes a group R or a hydrogen or hydroxyl group ;

or

(II) a siloxaneoxyalkylene block copolymer surfactant having a hydrophilic-lipophilic balance (HLB) of from 4 to 14.

Patentansprüche

1. Nichtwässriges flüssiges Reinigungsmittel, umfassend eine nichtwässrige flüssige Phase, 1 bis 70 Gewichtsprozent Feststoffteilchen und dispergierte Tröpfchen, umfassend Siliconpolymer und hydrophobe Teilchen, wobei die Tröpfchen eine Viskosität von wenigstens 5000 mPa·s bei 21 s^{-1} bei 25°C aufweisen, wobei die flüssige Phase weiterhin ein flüssiges nichtionisches Tensid umfaßt, mit der Maßgabe, daß das flüssige nichtionische Tensid nicht nur besteht aus

I. einem Polyorganosiloxan/Polyoxyalkylen-Copolymer, welches weder vollständig in einer wäßrigen Tensidlösung löslich ist, noch vollständig löslich ist in einem flüssigen verzweigten Polyhydrocarbylsiloxan-Material, das maximal zu 2mol% verzweigt ist, wobei wenigstens 50% aller vorhandenen Einheiten die allgemeine Formel $R_2-Si-O_{2/2}$ haben, worin R für eine monovalente Kohlenwasserstoffgruppe steht, die bis zu 24 Kohlenstoffatome aufweist und in der wenigstens 2 Kettenglieder die allgemeine Formel $R-Si-O_{3/2}$ oder $Si-O_{4/2}$ und alle anderen Kettenglieder die allgemeine Formel $R'_a-Si-O_{(4-a)/2}$ haben, wobei a einen Wert von 0 bis 3 annimmt und R' für eine Gruppe R , ein Wasserstoffatom oder eine Hydroxygruppe steht, oder

II. einem Siloxanoxyalkylen Block-Copolymer Tensid, das eine Hydrophilie-Lipophilie Balance (HLB) von 4 bis 14 aufweist.

2. Mittel nach Anspruch 1, wobei die Tröpfchen eine Viskosität von 5000 bis 60000 mPa·s bei 21 s^{-1} bei 25°C aufweisen.

3. Mittel nach Ansprüchen 1-2, wobei die hydrophoben Teilchen hydrophobes Siliciumdioxid sind.

4. Mittel nach Ansprüchen 1-3, wobei das Siliconpolymer Polydimethylsiloxan ist.

5. Mittel nach Ansprüchen 1-4, wobei die Tröpfchen in einer Menge von 0,1 bis 5,0 Gewichtsprozent des Mittels vorliegen.

6. Mittel nach Ansprüchen 1-5, wobei das Gewichtsverhältnis des Siliconpolymers zu den hydrophoben Teilchen 70:30 bis 99:1 ist.

7. Mittel nach Ansprüchen 1-6, wobei das Polymer ein Gemisch von Polydimethylsiloxan hoher Viskosität und Polydimethylsiloxan geringer Viskosität umfaßt.

8. Verfahren zur Herstellung eines nichtwässrigen flüssigen Reinigungsmittels, umfassend eine nichtwässrige flüssige Phase, 1 bis 70 Gewichtsprozent Feststoffteilchen und dispergierte Tröpfchen, wobei die Tröpfchen Siliconpolymer und hydrophobe Teilchen umfassen und eine Viskosität von wenigstens 5000 mPa·s bei 21 s^{-1} bei 25°C aufweisen, wobei die Feststoffteilchen mit der nichtwässrigen flüssigen Phase vermischt werden und gegebenenfalls vorher, während oder nach dem Mischen mit der nichtwässrigen flüssigen Phase vermahlen werden, wonach die Tröpfchen in der nichtwässrigen flüssigen Phase dispergiert werden, wobei die flüssige Phase weiterhin ein flüssiges nichtionisches Tensid umfaßt, mit der Maßgabe, daß das flüssige nichtionische Tensid nicht nur besteht aus

I. einem Polyorganosiloxan/Polyoxyalkylen-Copolymer, welches weder vollständig in einer wäßrigen Tensid-

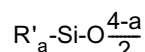
lösung löslich ist, noch vollständig löslich ist in einem flüssigen verzweigten Polyhydrocarbylsiloxan-Material, das maximal zu 2mol% verzweigt ist, wobei wenigstens 50% aller vorhandenen Einheiten die allgemeine Formel $R_2\text{-Si-O}_{2/2}$ haben, worin R für eine monovalente Kohlenwasserstoffgruppe steht, die bis zu 24 Kohlenstoffatome aufweist und in der wenigstens 2 Kettenglieder die allgemeine Formel $R\text{-Si-O}_{3/2}$ oder $\text{Si-O}_{4/2}$ und alle anderen Kettenglieder die allgemeine Formel $R'_a\text{-Si-O}_{(4-a)/2}$ haben, wobei a einen Wert von 0 bis 3 annimmt und R' für eine Gruppe R, ein Wasserstoffatom oder eine Hydroxygruppe steht, oder

II. einem Siloxanoxyalkylen Block-Copolymer Tensid, das eine Hydrophilie-Lipophilie Balance (HLB) von 4 bis 14 aufweist.

Revendications

1. Composition de nettoyage liquide non aqueuse comprenant une phase liquide non aqueuse, de 1 à 70% en poids de particules solides et de gouttelettes dispersées comprenant un polymère de silicone et des particules hydrophobes, lesdites gouttelettes ayant une viscosité au moins de 5000 mPa.s à 21 s^{-1} à 25°C , ladite phase liquide comprenant en outre un tensioactif non ionique liquide, à condition que le tensioactif non ionique liquide ne soit pas seulement composé :

(I) d'un copolymère de polyorganosiloxane et de polyoxyalkylène, qui n'est ni complètement soluble dans une solution aqueuse de tensioactif, ni complètement soluble dans un matériau liquide à base de polyhydrocarbylsiloxane ramifié ayant au maximum 2 moles % de ramification où au moins 50% de toutes les unités présentes ont la formule générale $R_2\text{-Si-O}_{2/2}$ dans laquelle R représente un groupe hydrocarboné monovalent ayant jusqu'à 24 atomes de carbone et où au moins 2 unités ont la formule générale $R\text{-Si-O}_{3/2}$ ou $\text{Si-O}_{4/2}$, toutes les autres unités ayant comme formule générale



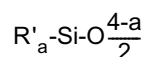
dans laquelle a vaut de 0 à 3 et R' représente un groupe R, un atome d'hydrogène ou un groupe hydroxy, ou

(II) un tensioactif formé par un bloc copolymère siloxanoxyalkylène ayant une balance hydrophilelipophile (HLB) comprise entre 4 et 14.

2. Composition selon la revendication 1, dans laquelle les gouttelettes ont une viscosité de 5000 à 60.000 mPa.s à 21 s^{-1} à 25°C .
3. Composition selon les revendications 1 et 2, dans laquelle les particules hydrophobes sont de la silice hydrophobe.
4. Composition selon les revendications 1 à 3, dans laquelle le polymère de silicone est le polydiméthylsiloxane.
5. Composition selon les revendications 1 à 4, dans laquelle les gouttelettes sont présentes en une quantité de 0,1 à 5,0% en poids de la composition.
6. Composition selon les revendications 1 à 5, dans laquelle le rapport pondéral du polymère de silicone aux particules hydrophobes est de 70:30 à 99:1.
7. Composition selon les revendications 1 à 6, dans laquelle le polymère comprend un mélange de polydiméthylsiloxane à viscosité élevée et un polydiméthylsiloxane à faible viscosité.
8. Procédé de préparation d'une composition détergente liquide non aqueuse comprenant une phase liquide non aqueuse, de 1 à 70% en poids de particules solides et de gouttelettes dispersées, lesdites gouttelettes comprenant un polymère de silicone et des particules hydrophobes et ayant une viscosité au moins de 5000 mPa.s à 21 s^{-1} à 25°C , dans lequel on mélange les particules solides avec la phase liquide non aqueuse et facultativement, on broie avant, pendant ou après le mélange avec la phase liquide non aqueuse, après quoi on disperse les gouttelettes dans la phase liquide non aqueuse, ladite phase liquide comprenant en outre un tensioactif non ionique liquide, à condition que le tensioactif non ionique liquide ne soit pas seulement composé :

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(I) d'un copolymère de polyorganosiloxane et de polyoxyalkylène, qui n'est ni complètement soluble dans une solution aqueuse de tensioactif, ni complètement soluble dans un matériau liquide à base de polyhydrocarbysiloxane ramifié ayant au maximum 2 moles % de ramification où au moins 50% de toutes les unités présentes ont la formule générale $R_2\text{-Si-O}_{2/2}$ dans laquelle R représente un groupe hydrocarboné monovalent ayant jusqu'à 24 atomes de carbone et où au moins 2 unités ont la formule générale $R\text{-Si-O}_{3/2}$ ou $\text{SiO}_{4/2}$, toutes les autres unités ayant comme formule générale



dans laquelle a vaut de 0 à 3 et R' représente un groupe R, un atome d'hydrogène ou un groupe hydroxy, ou

(II) un tensioactif formé par un bloc copolymère siloxaneoxyalkylène ayant une balance hydrophilelipophile (HLB) comprise entre 4 et 14.

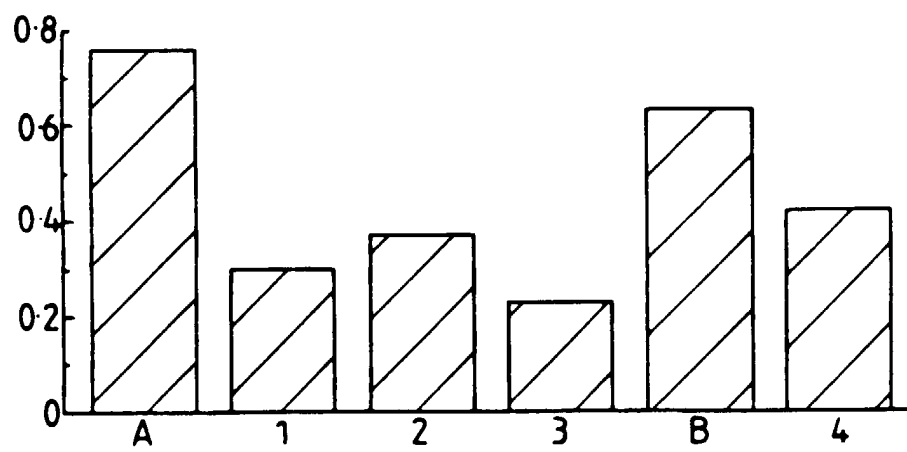


Fig: 1