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(54) Title: ALKALINE HARD SURFACE CLEANING AND DISINFECTING COMPOSITIONS		
(57) Abstract Hard surface cleaning and disinfecting compositions include a film-forming, organosilicone quaternary ammonium compound providing a protective layer for water and stain repellency.		

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5 **ALKALINE HARD SURFACE CLEANING AND DISINFECTING COMPOSITIONS**

The invention relates to alkaline hard surface cleaning and disinfecting compositions providing a protective layer for water and stain repellency.

10 Cleaning compositions are commercially important products and enjoy a wide field of utility in assisting in the removal of dirt and grime from surfaces, especially surfaces encountered in lavatories such as toilets, shower stalls, bathtubs, bidets, sinks, etc., as well as countertops, walls, floors, etc. In such lavatory environment, various forms of undesirable residues are known to form, particularly "soap scum stains". Soap scum stains are residues of fatty acid soaps
15 such as those are based on alkali salts of low fatty acids, which precipitate in hard water due to the presence of metal salts therein, leaving an undesirable residue upon such surfaces.

The prior art has suggested many compositions which are directed to the cleaning of such hard water and soap scum stains. ("Soap scum" is sometimes
20 referred to as "limescale" in Europe.) Many of these are acidic, aqueous compositions which include one or more deterative surfactants. A limited number of these compositions, in addition to a deterative benefit, also provide a germicidal or sanitizing effect to the hard surfaces being treated. While these acidic aqueous compositions are effective in the removal of hard water stains, they also may be
25 particularly detrimental to so-called "European porcelain" or "European enamel" hard surfaces. Such surfaces are known to be particularly sensitive to acidic compositions, especially acidic compositions characterized in having a low pH (less than pH=4). Thus, the use of acidic compositions are typically not compatible with such "European porcelain" or "European enamel" hard surfaces.

30 Few prior art compositions also suffer from the shortcoming in that they do not provide any significant long term cleaning or sanitizing benefit to the treated hard surfaces as they are easily rinsed away with water, and thus are not retained on the treated hard surface.

Accordingly, there is a real and continuing need in the art for improved hard surface treatment compositions which provide a cleaning or disinfecting benefit, (preferably both) and which form a film on the treated surface to provide a residual protective benefit. Desirably such compositions would be compatible for use on so-called "European porcelain" or "European enamel" hard surfaces.

According to a first aspect of the invention, there is provided an aqueous, alkaline hard surface cleaning composition which provides a cleaning benefit or disinfecting benefit (preferably both benefits) to a hard surface. The composition comprises the following constituents:

- (a) a film-forming, organosilicone quaternary ammonium compound;
- (b) at least one amine oxide surfactant;
- (c) at least one nonionic surfactant;
- (d) at least one organic solvent; and,
- (e) optionally, at least one amphoteric surfactant;
- (e) water.

wherein the aqueous compositions are at an alkaline pH, preferably a pH of 8 or greater, and wherein the aqueous compositions may be characterized as forming a film or surface coating which provides the benefit of water or stain repellency to the treated hard surface, or provides the benefit of residual disinfection to the treated hard surface, but preferably provides both benefits.

The compositions described above may include one or more further optional constituents including but not limited to further non-aqueous (organic) solvents, pH buffering agents, perfumes, perfume carriers, colorants, hydrotropes, germicides, fungicides, further antimicrobial compounds including quaternary ammonium compounds such as di(C₁-C₃alkyl)di(short chain alkyl) quaternary ammonium compounds, anti-oxidants, anti-corrosion agents, etc.

The compositions according to the invention are largely aqueous, and are readily pourable and pumpable. The preferred compositions all exhibit good storage stability.

Preferably the inventive compositions are highly alkaline in nature, and are at a pH of at least 8, at preferably at least 10 and higher, especially at a pH of at least 12.

Preferred embodiments of the invention form a film or surface coating on the treated hard surfaces. This film or surface coating provides the benefit of water or stain repellency to the treated hard surface.

According to a preferred embodiment of the invention, the inventive composition is also essentially free of conventional chelating agents such as ethylenediaminetetraacetic acid, N-hydroxyethylethylenediamine triacetic acid, nitrilotriacetic acid, diethylene triamine pentaacetic acid, and their water soluble salts, especially the alkali metal salts and particularly the sodium salts. The composition is also essentially free of gluconic acid, tartaric acid, citric acid, oxalic acid, and lactic acid.

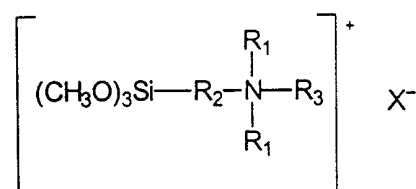
According to a second aspect of the invention, there is provided a process for cleaning or sanitization of hard surfaces, which process comprises the steps of providing the composition as outlined above, and applying an effective amount to a hard surface requiring such cleaning and/or sanitization.

The alkaline, aqueous compositions according to the invention comprise (a) a film-forming, organosilicone quaternary ammonium compound. Such compounds desirably also exhibit antimicrobial activity, especially on hard surfaces.

Specific examples of organosilicone quaternary ammonium salts that may be used in the compositions of this invention include organosilicone derivatives of the following ammonium salts: di-isobutylcresoxyethoxyethyl dimethyl benzyl ammonium chloride, di-isobutylphenoxyethoxyethyl dimethyl benzyl ammonium chloride, myristyl dimethylbenzyl ammonium chloride, myristyl picolinium chloride, N-ethyl morpholinium chloride, laurylisoquinolinium bromide, alkyl imidazolinium chloride, benzalkonium chloride, cetyl pyridinium chloride, coconut dimethyl benzyl ammonium chloride, stearyl dimethyl benzyl ammonium chloride, alkyl dimethyl benzyl ammonium chloride, alkyl diethyl benzyl ammonium chloride, alkyl dimethyl benzyl ammonium bromide, di-isobutyl phenoxyethoxyethyl trimethyl ammonium chloride, di-isobutylphenoxyethoxyethyl dimethyl alkyl ammonium chloride, methyl-dodecylbenzyl trimethyl ammonium chloride, cetyl trimethyl ammonium bromide, octadecyl dimethyl ethyl ammonium bromide, cetyl dimethyl ethyl ammonium bromide, octadec-9-enyl dimethyl ethyl ammonium bromide, dioctyl dimethyl

ammonium chloride, dodecyl trimethyl ammonium chloride, octadecyl trimethyl ammonium chloride, octadecyl trimethyl ammonium bromide, hexadecyl trimethyl ammonium iodide, octyl trimethyl ammonium fluoride, and mixtures thereof. Other water dispersible salts, such as the acetates, sulfates, nitrates and phosphates, are effective in place of the halides, but the chlorides and bromides are preferred. The silicone group is preferably substituted with alkyl ethers. Preferred alkyl ethers are short carbon chain ethers such as methoxy and ethoxy substituents.

Examples of particularly preferred film-forming, organosilicone quaternary ammonium compounds which find use in the present inventive compositions include those which may be represented by the structure:



wherein:

- R_1 and R_2 each independently represents short chain alkyl or alkenyl groups, preferably C_1 - C_8 alkyl or alkenyl groups;
 R_3 represents a C_{11} - C_{22} alkyl group; and
 X represents a salt forming counterion, especially a halogen.

Preferred short chain alkyl substituents for R_1 are methyl and ethyl. Preferred short chain alkyl substituents for R_2 are straight chain links of methylene groups consisting of from 1 to 4 members. Preferred R_3 substituents are straight chain links of methylene groups consisting of from 11 to 22 members. Preferred halogens for X are chloride and bromide. More preferably, both R_1 and R_2 are methyl.

A particularly preferred and commercially available film-forming, organosilicone quaternary ammonium compound useful in the inventive compositions is AEM® 5772 or AEM® 5700 (from Aegis Environmental Co., Midland, MI). Both of these materials are described as being 3-(trimethoxysilyl)propyloctadecyldimethylammonium chloride AEM® 5700 and is sold as a 42% by weight active solution of the compound in a water/methanol

mixture, while AEM® 5772 is sold as a 72% by weight active solution of the compound in a water/methanol mixture.

The film-forming, organosilicone quaternary ammonium compounds are desirably present in the inventive compositions in amounts of from 0.01 to 1.0% by weight, preferably in amounts of from 0.05 to 0.9%wt., and most preferably from 0.1 to 0.7% by weight, based on the total weight of the aqueous composition of which it forms a part.

The compositions of the invention also contain (b) at least one amine oxide surfactant compound, which is compatible with the (a) film-forming, organosilicone quaternary ammonium compound.

Amine oxide compounds (b) which are useful in the compositions of the invention are known to the art. One general class of useful amine oxides include alkyl di(lower alkyl) amine oxides in which the alkyl group has about 10-20, and preferably 12-16 carbon atoms, and can be straight or branched chain, saturated or unsaturated. The lower alkyl groups include between 1 and 7 carbon atoms. Examples include lauryl dimethyl amine oxide, myristyl dimethyl amine oxide, dimethyl cocoamine oxide, dimethyl (hydrogenated tallow) amine oxide, and myristyl/palmityl dimethyl amine oxide. Further amine oxides include alkyl di(hydroxy lower alkyl) amine oxides in which the alkyl group has about 10-20, and preferably 12-16 carbon atoms, and can be straight or branched chain, saturated or unsaturated. Examples are bis(2-hydroxyethyl) cocoamine oxide, bis(2-hydroxyethyl) tallow amine oxide, and bis(2-hydroxyethyl) stearylamine oxide. Yet further useful amine oxides include alkylamidopropyl di(lower alkyl) amine oxides in which the alkyl group has about 10-20, and preferably 12-16 carbon atoms, and can be straight or branched chain, saturated or unsaturated. Examples are cocoamidopropyl dimethyl amine oxide and tallowamidopropyl dimethyl amine oxide.

Suitable amine oxides, including many of those recited above, include those which are presently commercially available and include those under the trade name Ammonyx® (Stepan Co., Chicago IL), as well as Barlox® (Lonza Inc., Fairlawn NJ)

With respect to the amine oxides, preferred are the alkyl di(lower alkyl) amine oxides in which the alkyl group has about 8-16 carbon atoms. Exemplary and

preferred amine oxide compounds include N-alkyldimethylamine oxides, particularly octyldimethylamine oxides as well as lauryldimethylamine oxide.

The amine oxide constituent forms from 0.01 - 2%wt. of the inventive compositions, preferably comprise form 0.01 - 1%wt.

- 5 The compositions of the present invention further include (c) a nonionic surfactant. Suitable nonionic surfactants include, inter alia, condensation products of alkylene oxide groups with an organic hydrophobic compound, such as an aliphatic compound or with an alkyl aromatic compound. One example of such a nonionic surfactant is the condensation product of one mole of an alkylphenol
10 having an alkyl group containing from 6 to 12 carbon atoms with from about 5 to 25 moles of an alkylene oxide. Another example of such a nonionic surfactant is the condensation product of one mole of an aliphatic alcohol which may be a primary, secondary or tertiary alcohol having from 6 to 18 carbon atoms with from 1 to about 10 moles of alkylene oxide. Preferred alkylene oxides are ethylene
15 oxides or propylene oxides or mixtures thereof.

- Preferred nonionic surfactants include primary and secondary linear and branched alcohol ethoxylates based on C₁₀-C₁₆ alcohols and having from 3 to 10 moles of ethoxylation per mole of alcohol. Particularly preferred nonionic surfactants are C₁₁ linear primary alcohol ethoxylates averaging about 9 moles of
20 ethylene oxide per mole of alcohol. These surfactants are available, for example, under the commercial name of Neodol 1-9, (from Shell Chemical Company, Houston, TX) , or in the Genapol® series of linear alcohol ethoxylates, particularly Genapol® 26-L-60 or Genapol® 26-L-80 (from Clariant Corp., Charlotte, NC).

- 25 It is to be understood that nonionic surfactants other than those described above may also be used. For example, these include: secondary C₁₂ to C₁₅ alcohol ethoxylates, including those which have from about 3 to about 10 moles of ethoxylation which are available in the Tergitol® series of nonionic surfactants (Union Carbide Corp., Danbury, CT), particularly those in the Tergitol® "15-S-"
30 series. Further exemplary nonionic surfactants include linear primary C₁₁ to C₁₅ alcohol ethoxylates, including those which have from about 3 to about 10 moles of ethoxylation. Such are available in the Neodol® series of nonionic surfactants (Shell Chemical Co.)

The nonionic surfactants comprise 0.05 to 1.5% by weight of the compositions, preferably from 0.1 to 1.0% by weight, and most preferably from 0.15 to 0.5% by weight.

The compositions of the invention also comprise (d) an organic solvent constituent. Preferred solvents include water miscible alcohols, amines, amides, esters and ethers. Mixtures of one or more of these organic solvents can also be used.

Preferred as solvents in this invention are the glycol ethers having the general structure R_a-O-R_b-OH , wherein R_a is an alkoxy of 1 to 20 carbon atoms, or aryloxy of at least 6 carbon atoms, and R_b is an ether condensate of propylene glycol and/or ethylene glycol having from 1 to 10 glycol monomer units. These materials include those available in the DOWANOL™ glycol ether series (ex. Dow Chemical Co.), or the CARBITOL® series (ex. Union Carbide Corp.). More preferably employed as the (d) organic solvent constituent is one or more solvents of the group consisting of: propylene glycol n-propyl ether, dipropylene glycol n-propyl ether, propylene glycol n-butyl ether, dipropylene glycol n-propyl ether, ethylene glycol n-butyl ether, diethylene glycol n-butyl ether, and mixtures thereof. Most preferably, the solvent is a diethylene glycol n-butyl ether which is commercially available as DOWANOL DB. In certain preferred embodiments the (d) organic solvent constituent consists solely of diethylene glycol n-butyl ether.

The compositions of the invention contain from 0.1 to 10% by weight of the organic solvent constituent, preferably from 1 to 8% by weight, more preferably from 2 to 7% by weight.

Optionally, but in certain cases desirably, the inventive compositions include (e) at least one amphoteric surfactant. By way of example, these include the salts of higher alkyl beta-amino propionic acids, e.g., sodium N-lauryl beta-alanine; the higher alkyl substituted betaines, such as lauryl dimethylammonium acetate; as well as amphoteric surfactants of the imidazoline type exemplified by the disodium salt of 1-(2-hydroxyethyl)-1-(carboxymethyl)-2-(hendecyl)-4,5-dihydroimidazolinium hydroxide. An exemplary preferred amphoteric surfactant is lauramidopropionic acid, which is commercially available in the DERIPHAT series (ex Henkel) or MACKAM series (ex McIntyre Group

Inc.) of amphoteric surfactants. When present, they may comprise up to 5%wt. of the inventive compositions.

The compositions are largely aqueous in nature, and comprise as a further necessary constituent (f) water. Water is added to order to provide to 100% by weight of the compositions of the invention, and comprises at least 80% of the compositions, preferably at least 85% of the compositions. The water is preferably deionized water.

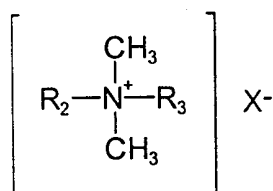
As discussed previously, the inventive compositions may comprise one or more conventional optional additives. Of course, these must be compatible with the other constituents present in the compositions. By way of non-limiting example, these include: pH adjusting agents and pH buffers including organic and inorganic salts; non-aqueous solvents, perfumes and perfume carriers; optical brighteners; coloring agents such as dyes and pigments; opacifying agents; hydrotropes; antifoaming agents; viscosity modifying agents such as thickeners; enzymes; anti-spotting agents; anti-oxidants; and anti-corrosion agents. These ingredients may be present in any combination and in any suitable amount that is sufficient for imparting the desired properties to the compositions, but it is to be understood that, in accordance with preferred embodiments of the invention, the inventive compositions are essentially free of conventional chelating agents. These one or more conventional optional additives, when present, should be present in minor amounts, preferably in total comprising less than about 5% by weight of the compositions, and desirably less than about 3%wt.

The compositions of the invention show excellent efficacy at high pHs. The inventive compositions desirably have a pH of at least 10 or higher, more desirably a pH of 12 or higher. This may be attained by the use of basic pH-adjusting constituents, which may be any material which is effective in adding to the alkalinity of the inventive compositions. By way of non-limiting examples, useful pH adjusting constituents include inorganic bases such as alkali metal salts of hydroxides, carbonates, and other suitable ions. Preferably, the pH-adjusting constituents are chosen from sodium or potassium hydroxide and sodium or potassium carbonate. Other inorganic bases not specifically elucidated here may also be used. The basic pH-adjusting constituent is desirably present in the compositions of the invention from about 0.2 to 10% by weight, preferably from

0.5 to 7%, most preferably from 1 to 4% by weight based on the total weight of the compositions.

As an optional constituent, it may be desirable to include one or more further compounds to provide an additional sanitizing or antimicrobial effect.

- 5 Exemplary antimicrobial compounds including quaternary ammonium compounds such as di(C₁-C₃alkyl)di(short chain alkyl) quaternary ammonium compounds. These further quaternary ammonium compounds include those which have the structural formula:



- 10 wherein R₂ and R₃ are the same or different C₈-C₁₂alkyl groups, or R₂ is C₁₂₋₁₆alkyl, C₈₋₁₈alkylethoxy, C₈₋₁₈alkylphenylethoxy and R₃ is benzyl, and X is a halide, for example chloride, bromide or iodide, or methosulfate. The alkyl groups recited in R₂ and R₃ may be straight-chained or branched, but are preferably substantially
15 linear.

- Particularly useful quaternary germicides include compositions which include a single quaternary compound, as well as mixtures of two or more different quaternary compounds. Particularly useful quaternary germicides include which are presently commercially available under the tradenames
20 BARDAC, BARQUAT, BTC, LONZABAC and HYAMINE, available from Stepan Co. (Chicago IL) or Lonza Inc. (Basle, CH) Especially preferred compounds are described in the Examples.

- When present, these further quaternary ammonium compounds providing additional sanitizing or antimicrobial effects may be present in any effective
25 amount, and when present are usually present in an amount of from 0.001 – 2%wt., more preferably from 0.01 – 1%wt., based on the total weight of the composition. Most preferably, when such further quaternary ammonium compounds are included, they are present in an amount of at least about 200 parts per million in the aqueous compositions of which they form a part.

- 30 According to a particularly preferred embodiment of the invention, there is provided an aqueous, alkaline hard surface cleaning composition which provides

a cleaning benefit or disinfecting benefit (preferably both benefits) to a hard surface. The composition comprises, (but preferably consists essentially of,) the following constituents:

- 5 (a) 0.01 to 1.0%wt. of a film-forming, organosilicone quaternary ammonium compound;
- (b) 0.05 to 5%wt. of at least one amine oxide surfactant;
- (c) 0.05 to 1.5%wt. of at least one nonionic surfactant;
- (d) 0.1 to 10%wt. of at least one organic solvent;
- (e) 0 - 5%wt. of amphoteric surfactant;
- 10 (f) 80 - 100%wt. water; and,
- (g) 0 - 5%wt. of one or more optional constituents;

wherein the aqueous compositions are at an alkaline pH, preferably at a pH of at least 10, and wherein the aqueous compositions may be characterized as forming a film or surface coating which provides the benefit of water or stain repellency to the treated hard surface, or provides the benefit of residual disinfection to the

15 the treated hard surface, but preferably provides both benefits.

The aqueous compositions according to the invention are desirably provided as a ready to use product which may be directly applied to a hard surface. Hard surfaces which are to be particularly denoted are lavatory fixtures and lavatory appliances (toilets, bidets, shower stalls, bathtubs and bathing

20 appliances), wall and flooring surfaces especially those which include refractory materials and the like. Further hard surfaces which are particularly denoted are those associated with kitchen environments and other environments associated with food preparation.

25 The aqueous compositions according to the invention are particularly useful in the treatment of hard surfaces wherein soap scum is prone to occur, particularly hard surfaces associated with lavatories, including lavatory fixtures and appliances.

The compositions according to the invention can be desirably provided as ready to use products in manually operated spray dispensing

30 containers, or may be supplied as aerosol type products discharged from a pressurized aerosol container. Known art propellants such as liquid propellants

based on chlorofluorocarbons or propellants of the non-liquid form, i.e., pressurized gases, including carbon dioxide, air, nitrogen, as well as others, may be used.

Whereas the compositions of the present invention are intended to be used in the types of liquid forms described above, nothing in this specification shall be understood as to limit the use of said compositions with a further amount of water to form a cleaning solution.

The following examples below illustrate exemplary and preferred formulations of the concentrate composition according to the instant invention. It is to be understood that these examples are presented by means of illustration only and that further useful formulations fall within the scope of this invention and the claims may be readily produced by one skilled in the art without deviating from the scope and spirit of the invention.

Throughout this specification and in the accompanying claims, weight percents of any constituent are to be understood as the weight percent of the active portion of the referenced constituent, unless otherwise indicated.

Examples

The following examples illustrate the formulation and performance of various compositions of the invention.

Exemplary formulations illustrating certain preferred embodiments of the inventive compositions and described in more detail in Table 1 below were formulated generally in accordance with the following protocol. The weight percentages indicated the "as supplied" weights of the named constituent.

Into a suitably sized vessel, a measured amount of water was provided after which the constituents were added in no specific or uniform sequence, thus indicating that the order of addition of the constituents was not critical. All of the constituents were supplied at room temperature, and any remaining amount of water was added thereafter. Certain of the nonionic surfactants if gels at room temperature were first preheated to render them pourable liquids prior to addition and mixing. Mixing of the constituents was achieved by the use of a mechanical stirrer with a small diameter propeller at the end of its rotating shaft. Mixing, which generally lasted from 5 minutes to 120 minutes was maintained until the particular exemplary formulation appeared to be homogeneous. The exemplary

compositions were readily pourable, and retained well mixed characteristics (i.e., stable mixtures) upon standing for extended periods. The compositions of the example formulations are listed on Table 1.

Table 1						
	Ex.1	Ex.2	Ex.3	Ex.4	Ex.5	Ex.6
AEM 5700 (42%)	0.48	0.36	0.24	0.24	0.60	0.36
Mackamine C-8 (40%)	1.22	1.22	1.22	1.22	1.22	--
Ammonyx LO (30%)	--	--	--	--	--	2.50
Neodol 1-9 (100%)	0.26	0.26	0.26	0.26	0.26	--
Genapol 26-L-80	--	--	--	--	--	0.60
Dowanol DB (100%)	5.99	5.99	5.99	5.99	5.99	5.99
sodium carbonate (100%)	2.25	2.25	2.25	2.25	2.25	2.25
sodium hydroxide (25%)	0.426	0.426	0.426	0.426	0.426	0.45
fragrance	--	--	--	--	--	--
DI water	to 100	to 100	to 100	to 100	to 100	to 100
pH of formulation:	12.34	12.38	12.44	12.39	12.23	12.50

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The identity of the constituents of Table 1 above are described in more detail on Table 2, below, including the "actives" percentage of each.

TABLE 2	
Ingredient (% weight active)	
AEM 5700 (42%);	organosilicone quaternary ammonium compound from Aegis Co.
Mackamine C-8 (40%)	octyl amine oxide surfactant surfactant from McIntyre Group LTD
Ammonyx LO (30%)	lauryl dimethyl amine oxide surfactant
Neodol 1-9 (100%)	nonionic alcohol ethoxylate surfactant from Shell Chemical
Genapol 26-L-80	linear alcohol ethoxylate from Clariant Corp.
Dowanol DB (100%)	diethylene glycol n-butyl ether from Eastman Chemical Co.
sodium carbonate (100%)	sodium carbonate from General Chemical Co., Morristown NJ
sodium hydroxide (25%)	sodium hydroxide in an aqueous solution from Aldrich Chem. Co.
fragrance	proprietary composition
DI water	deionized water

10 The formulations described in Table 1 were subjected to one or more of the following evaluations.

Cleaning Efficacy

The cleaning efficacy of each tested formulations was evaluated in order to determine their efficacy in the removal soap scum.

Soap Scum (Limescale) Cleaning Test

For the performance of this test, the following materials were utilized. As substrate samples: standard square glazed black ceramic tile, measuring 10.8 cm by 10.8 cm. As cleaning medium, a standard cellulose sponge. If the sponge was supplied with a surfactant or other entrained material, such were first removed by washing with warm water, either by hand or by machine, followed by complete drying of the sponge. As a test shampoo, a simple moderate-cleaning type containing alkyl ethoxysulfates may be used. An exemplary shampoo composition is listed in the CSMA DCC-16 protocol.

This test is described generally as follows:

Soil Preparation

A "parent" soil is made, based on the following formulation:

<u>"Parent" soil</u>	<u>% w/w</u>
bar soap	3.90
shampoo	0.35
clay	0.06
artificial sebum	0.15
hard water	95.54

The parent soil was produced according to the following steps: First, the bar soap was shaved into a suitable beaker. Afterward the remaining constituents were added in the order given above and stirred with three-blade propeller mixer. Next, the contents of the beaker was heated to 45-50°C and mixed until a smooth, lump-free suspension was achieved. This usually required about two hours with moderate agitation. Subsequently, the contents of the beaker were filtered through a Buchner funnel fitted with Whatman #1 filter paper or equivalent. The filtrate was then resuspended in clean, deionized water, using the same amount of water used to make the soil, and this was filtered again. The (re-filtered) filtrate was uniformly dried overnight at 45°C to form a filter cake. Thereafter, the filter cake was pulverized and was suitable for immediate use, or may be stored in a sealed container for up to six months.

Substrate preparation:

The test substrates (tiles) were prepared in the following manner: each tile was thoroughly washed (using a commercially available hand dishwashing detergent, Dove®) and scrubbed using a non-metallic scouring pad (such as a

Chore Boy® Long Last scrubbing sponge). The washed tiles were then permitted to dry in an oven at 40.5°C overnight, then withdrawn and allowed to cool to room temperature (approx. 20°C) before being provided with the standardized “hard water” test soil. It is to be noted that for each test, new tiles were utilized, namely, the tiles were not reused.

In preparation for supplying the tiles with an amount of the test soil, a test soil was prepared based on the following formulation:

<u>Test soil:</u>	<u>%w/w</u>
“parent” soil	4.50
hard water	9.0
hydrochloric acid (0.1N)	0.77
acetone	85.73

The test soil was produced according to the following steps: The constituents indicated were introduced into a clean beaker, with the acetone being added prior to the water, and the ‘parent’ soil being added last. The contents of the beaker were mixed using a standard three blade laboratory mixer until the contents formed a uniform mixture, and the color changed from white to gray. This typically required 20-40 minutes, during which time the beaker was covered as much as possible to avoid excessive solvent loss. Next, a suitable quantity of the contents of the test soil from the beaker was provided to an artist’s airbrush while the beaker was swirled to ensure a soil uniformity. (If testing required more than one day, a fresh amount of test soil was prepared daily and used for that day’s testing.)

Soil was applied to a number of clean, dry tiles placed into rows and columns in preparation for depositing of the test soil. The airbrush was operated at 40 psi, and the test soil was sprayed to provide a visually uniform amount of soil onto the tiles. (Uniform soil suspension during application was maintained by continuous brush motion and/or swirling of test soil in the airbrush.) In this manner, approximately 0.10g-0.15g test soil were applied per tile.

The tiles were then allowed to air dry for approximately 30 minutes, during which time the a laboratory hotplate was preheated to approximately 320°C. Each tile was sequentially placed on the hotplate until the test soil began to melt, thereby “aging” the test soil. The melting of the test soil was observed carefully, and each tile was removed shortly before the soil began to coalesce into

large droplets. This process was repeated for each tile, allowing the hotplate to recover to 320°C between tiles. Subsequently each tile was permitted to cool for at least about 30 minutes.

Cleaning Evaluation

5 To evaluate cleaning, a treated test tile was placed in a Gardner Apparatus and secured. A dry 10 cm by 7.6 cm sponge was first moistened with 100 g of tap water, and the excess wrung out from the sponge. The sponge was then fitted into a suitably sized holder in the Gardner Apparatus. A 4-5 gram aliquot of a test formulation was then deposited directly onto the soiled surface of a tile, and
10 allowed to contact the tile for 15 seconds. Thereafter, the Gardner Apparatus was cycled for from 3 – 6 strokes. The tile was then rinsed with tap water, and dried with compressed air from an airbrush compressor. This test was repeated several times for each formulation, using new treated test tile for each evaluation.

The tested tiles were evaluated by either reflective means, i.e., using a 60
15 degree angle reflectometer, (BYK-Gardner Co.) to measure the reflectance of the reference and treated tiles, or by objective means wherein a group of persons evaluated a set of tiles and provided an evaluation of the visual appearance of the tested tiles.

According to the reflective means, the percentage of hard water soil
20 removal was determined utilizing the following equation:

$$\% \text{ Removal} = \frac{RC - RS}{RO - RS} \times 100$$

where

25 RC = Reflectance of tile after cleaning with test product

RO = Reflectance of original soiled tile

RS = Reflectance of soiled tile

For each tile, a number of readings were taken and the results averaged to provide a median reading for each tile.

30 According to the objective means, the soil removal was visually examined by a minimum of 20 independent judges, who evaluated each of a set of tested tiles. A clean substrate and soiled but untreated substrate are used as references. Soil removal was rated as follows:

<u>Rating:</u>	<u>Description of rating:</u>
0	no soil removed, or minimal soil removed
10	approximately 10% soil removed
20	approximately 20% soil removed
30	approximately 30% soil removed
40	approximately 40% soil removed
50	approximately 50% soil removed
60	approximately 60% soil removed
70	approximately 70% soil removed
80	approximately 80% soil removed
90	approximately 90% soil removed
100	all soil removed

The tested tiles were evaluated, and the results are indicated on the Table 3, below.

5

Table 3	
	Soap Scum (Limescale)
Ex.1	80 – 90
Ex.2	80 – 90
Ex.3	80 – 90
Ex.4	80 – 90
Ex.5	80 – 90

Surface Protection

The surface repellency of treated tiles was evaluated by determining the contact angle of water on treated tile. The contact angle was determined utilizing a Kruss Goniometer, and the results were evaluated using a computer program titled "Contact Angle Measurement System G40 v.1.32-US (commercially available from Hewlett Packard Co.). On a test substrate, four readings were taken of the contact angles of a droplet of water and the average of these four readings indicated an angle of 75 degrees. This is indicative of the presence of a hydrophobic film on the surface of the treated tile.

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Evaluation of Antimicrobial Efficacy:

Formulation described in Table 1 above were evaluated in order to evaluate their antimicrobial efficacy against *Staphylococcus aureus* (Gram positive type pathogenic bacteria) (ATCC 6538), *Salmonella choleraesuis* (Gram negative type pathogenic bacteria) (ATCC 10708), and *Pseudomonas aeruginosa* (ATCC 15442). The testing was performed in accordance with the protocol of the Association of Official Analytical Chemists; "Germicidal Spray Test".

As is appreciated by the skilled practitioner in the art, the results of the AOAC Germicidal Spray Test indicates the number of test substrates wherein the tested organism remains viable after contact for 10 minutes with a test disinfecting composition / total number of tested substrates (cylinders) evaluated in accordance with the AOAC Germicidal Spray Test. Thus, a result of "0/30" indicates that, of 60 test substrates bearing the test organism and contacted for 10 minutes in a test disinfecting composition, 0 test substrates had viable (live) test organisms at the conclusion of the test. Such a result is excellent, illustrating the excellent disinfecting efficacy of the tested composition.

Results of the antimicrobial testing are indicated on Table 4, below. The reported results indicate the number of test cylinders with live test organisms/number of test cylinders tested for each example formulation and organism tested.

Table 4 - Antimicrobial Efficacy

<u>Example Formulation</u>	<i>Staphylococcus aureus</i>	<i>Salmonella choleraesuis</i>	<i>Pseudomonas aeruginosa</i>
Ex. 6	0/30	0/30	0/30

As may be seen from the results indicated above, the compositions according to the invention provide excellent cleaning benefits to hard surfaces, including hard surfaces with difficult to remove soap scum stains and exhibit excellent antimicrobial efficacy of these compositions against known bacteria commonly found in bathroom, kitchen and other environments.

Claims:

1. An alkaline aqueous hard surface treatment composition comprising:
 - (a) a film-forming, organosilicone quaternary ammonium compound;
 - 5 (b) at least one amine oxide surfactant;
 - (c) at least one nonionic surfactant;
 - (d) at least one organic solvent; and,
 - (e) optionally, at least one amphoteric surfactant;
 - (e) water;
- 10 wherein the compositions may be characterized as forming a film or surface coating which provides the benefit of water or stain repellency to the treated hard surface, or may be characterized in providing the benefit of residual disinfection to the treated hard surface.
- 15 2. An aqueous hard surface treatment composition of claim 1, further comprising from 1 to 20% by weight of at least one alkaline pH-adjusting agent.
3. An aqueous hard surface treatment composition of claim 1 wherein the
 - 20 organosilicone quaternary ammonium compound is a compound of the following formula
$$\left[\begin{array}{c} \text{R}_1 \\ | \\ (\text{CH}_3\text{O})_3\text{Si}-\text{R}_2-\text{N}-\text{R}_3 \\ | \\ \text{R}_1 \end{array} \right]^+ \text{X}^-$$

wherein R₁ and R₂ are C₁ to C₃ alkyl, R₃ is C₁₁ to C₂₂ alkyl, and X is a halogen.
- 25 4. The aqueous hard surface treatment composition according to claim 3 wherein the organosilicone quaternary ammonium compound is 3-(trimethoxysilyl) propyloctadecyldimethylammonium chloride.

5. The aqueous hard surface treatment composition according to claim 1 wherein the amine oxide surfactant is a C₆-C₁₂ amine oxide.
6. The aqueous hard surface treatment composition according to claim 5 wherein the amine oxide surfactant is a C₈ amine oxide.
7. The aqueous hard surface treatment composition according to claim 1 wherein the nonionic surfactant is an alcohol ethoxylate.
- 10 8. An aqueous hard surface cleaning composition of claim 1 which comprises up to 5%wt. of an amphoteric surfactant.
9. An alkaline aqueous hard surface cleaning composition of claim 1 comprising:
 - 15 (a) 0.01 to 1.0%wt. of a film-forming, organosilicone quaternary ammonium compound;
 - (b) 0.05 to 5%wt. of at least one amine oxide surfactant;
 - (c) 0.05 to 1.5%wt. of at least one nonionic surfactant;
 - (d) 0.1 to 10%wt. of at least one organic solvent;
 - 20 (e) 0 - 5%wt. of amphoteric surfactant;
 - (f) 80 - 100%wt. water; and,
 - (g) 0 - 5%wt. of one or more optional constituents.
10. A composition substantially as described with reference to the Examples.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 99/02616

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C11D11/00 C11D3/16 C11D3/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	US 5 885 951 A (EDWIN R. LODER) 23 March 1999 (1999-03-23) claims 1,2,4-6	1,3,4,9, 10
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A	EP 0 129 980 A (PROCTER & GAMBLE) 2 January 1985 (1985-01-02) claims 1,4,5,9	1-5,8-10
A	WO 92 14810 A (S.C. JOHNSON & SON) 3 September 1992 (1992-09-03) page 8, last paragraph -page 9, paragraph 2 page 10, paragraph 2 page 22, paragraph 1	1,3,4,7, 8



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

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INTERNATIONAL SEARCH REPORT

information on patent family members

International Application No

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