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(54) Title: LAUNDRY DETERGENT COMPOSITIONS COMPRISING AMINO SILICONE ANTIFOAM AGENT

(57) Abstract: Disclosed are laundry detergent compositions for machine or hand wash having an modified amino silicone based antifoam. The compositions produce higher foam during the wash cycle, and highly reduced or practically nil foam after two rinses. The compositions help conserve a large amount of water that gets used for rinsing clothes.

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LAUNDRY DETERGENT COMPOSITIONS COMPRISING AMINO SILICONE ANTIFOAM AGENT

FIELD OF INVENTION

The present invention relates to laundry detergent compositions having delayed-release amino silicone antifoam.

BACKGROUND ART

In a typical hand washing process of domestic laundry, water is taken in a bucket or tubs. Thereafter, desired quantity of detergent powder is added to the water, followed by hand-whisking for about 2 to 3 minutes. Soiled clothes are then soaked in the water for about 10 to 30 minutes, or even longer if desired. After the soaking period is over, the clothes are rubbed by hands or by an implement, such as a brush, to scrub off dirt.

When clothes are rinsed, the dirty water is drained out and excess dirty water is removed by squeezing the fabrics. Fresh water is used for each rinsing cycle, which is generally repeated four to five times or until most of the foam has disappeared.

When the consumers see lower foam or no foam during the washing cycle, it leads them to believe that the detergent is not as active as it should be. Consumers also believe that there should be minimal, or preferably no foam after rinsing the clothes. There has been increasing preference for detergent products which require lesser amount of water for rinsing.

Attempts have been made in the past to provide such products. In a concept, known as single rinse concept; a de-foamer product containing a fabric conditioner is added during the rinse cycle. Such products condition and soften the clothes and also reduce the foam.

Fabric conditioners are considered to be premium laundry products, and consumers who use detergent powders in domestic hand-wash laundry process, generally cannot afford fabric conditioners.

Considering the amount of water that gets wasted in the rinse process, there is a need for detergent compositions which generate higher amount of foam during the washing cycle, but at the same time, which exhibits enough antifoaming activity during the rinsing cycle, so that one or two rinses are sufficient.

US 4637890 A (Procter & Gamble, 1987) discloses a detergent composition that acts as defoamer in the rinsing cycle. The composition has suds/foam controlling prills having fatty acid soap, quaternary ammonium salt and silicone fluid. The prills dissolve at high pH (from 9 to 10.5) but are not active at the pH. Prills become active at lower pH during the rinse cycle, to suppress suds.

US 4894117 A (Colgate Palmolive Co, 1990) discloses a composition of agglomerated granules for the delayed release of antifoaming agent in laundry compositions. The antifoam is based on silicone, which is adsorbed on a powdered water soluble carrier selected from modified cellulose carriers which are subsequently agglomerated into granular form by mixing in presence of a solvent for the carrier.

Detergent powders containing improved delayed-release antifoam agents are desired.

We have determined that a modified amino silicone antifoam shows improved delayed-release action when used in detergent powders.

SUMMARY OF THE INVENTION

According to a first aspect of the invention there is provided a laundry detergent composition for machine or hand washing comprising:

(i) a modified amino silicone antifoam fluid of the formula

$\mathbf{X}R_2Si(OSi\mathbf{A}R)_n(OSiR_2)_mOSiR_2\mathbf{X}$ (I)

where

A is an amino radical of the formula

$-R^1-[NR^2-R^3-]_xNR^2{}_2$ or the protonated amino forms of the amino radical A

X is **R** or a polyoxyalkylen group **G** of the formula $-R^4-(O-R^5)_y-O-R^6$

R is a monovalent hydrocarbon radical having from 1 to 18 carbon atoms,

$\mathbf{R^1}$ is a divalent C_1-C_{10}-alkylen radical, preferably a radical of the formula $-CH_2CH_2CH_2-$,

$\mathbf{R^2}$ is a hydrogen atom or a C_1-C_4-alkyl radical, preferably a hydrogen atom,

$\mathbf{R^3}$ is a divalent C_1-C_{10}-alkylen radical, preferably a radical of the formula $-CH_2CH_2-$,

$\mathbf{R^4}$ is a divalent C_1-C_{10}-alkylen, preferably a radical of the formula $-CH_2CH_2CH_2-$,

$\mathbf{R^5}$ is a C_1-C_4-alkylen radical, preferably a radical of the formula $-CH_2CH_2-$ or $-CH_2CH_2(CH_3)-$ or mixtures of them,

$\mathbf{R^6}$ is a hydrogen atom or an C_1-C_4-alkyl radical, preferably a hydrogen atom or a methyl radical, more preferably a hydrogen atom,

n is an integer from 1 to 6, preferably from 1 to 3,

m is an integer from 1 to 200, preferably from 1 to 80,

x is 0 or 1 and

y is an integer from 5 to 20, preferably from 5 to 12,

with the provision that on average from 30 to 60 mol%, preferably 50 mol%, of radicals **X** are polyoxyalkylen groups **G**; and,

(ii) usual laundry detergent ingredients.

Preferably the amino radical (A) is selected from $-(CH_2)_3NH_2$; $-(CH_2)_3NH(CH_2)_2NH_2$; $-(CH_2)_3NHC_6H_{11}$; $-(CH_2)_3NH(CH_2)_2NHC_6H_{11}$; $-(CH(CH_3)CH_2CH_2)NH_2$; $-(CH(CH_3)CH_2CH_2)NH(CH_2)NH_2$; $-(CH_2)NH_2$; $-(CH_2)NH(CH_2)_2NH_2$;

$-(CH(CH_3)CH_2CH_2)NHC_6H_1$; $-(CH(CH_3)CH_2CH_2)NH(CH_2)NHC_6H_1$;
$-(CH_2)NHC_6H_{11}$; $-(CH_2)NH\ (CH_2)_2\ NHC_6H_{11}$ and acylated amine of the amino radicals.

The most preferred radical is selected from $-(CH_2)_3NH_2$ or $-(CH_2)_3\,NH\ (CH_2)_2NH_2$.

Preferably the amine number of the modified silicone antifoam fluid of formula (I) is from 0.6 mg of KOH/g of polymer to 2.0 mg of KOH/ g of polymer of the silicone fluid of formula (I), and more preferably from 1.0 to 1.5 mg of KOH/ g of polymer of the silicone fluid.

Preferably **X** involving polyoxyalkylen radical group **G** is $(CH_2)_3$-$(OC_2H_4)_y$-O-R^6; $-(CH(CH_3)CH_2CH_2)$-$(OC_2H_4)_y$-O-R^6; $-(CH_2)_3$-$(OC_3H_6)_y$-O-R^6; $-(CH(CH_3)CH_2CH_2)$-$(OC_3H_6)_y$-O-R^6; $-(CH_2)$-$(OC_2H_4)_y$-O-R^6; $-CH_2$-$(OC_2H_4)_y$-O-R^6; $-(CH_2)$-$(OC_3H_6)_y$-O-R^6; $-CH_2$-$(OC_3H_6)_y$-O-R^6. The most preferred polyoxyalkylen radical is $-(CH_2)_3$-$(OC_2H_4)_y$-O-R^6, where R^6 and y have the meanings described earlier.

Examples of hydrocarbon R are alkyl radicals, such as the methyl, ethyl, n-propyl, isopropyl, 1-n-butyl, 2-n-butyl, isobutyl, tert-butyl, n-pentyl, isopentyl, neopentyl and tert-pentyl radical, hexyl radicals, such as the n-hexyl radical, heptyl radicals, such as the n-heptyl radical, octyl radicals, such as the n-octyl radical and isooctyl radicals, such as the 2,2,4-trimethylpentyl radical, nonyl radicals, such as the n-nonyl radicals, decyl radicals, such as the n-decyl radical, dodecyl radicals, such as the n-dodecyl radical, and octadecyl radicals, such as the n-octadecyl radical; alkenyl radicals such as the vinyl and ally radical; cycloalkyl radicals, such as the cyclopentyl, cyclohexyl, cycloheptyl and methylcyclohexyl radicals; aryl radicals, such as the phenyl, naphthyl, anthryl and phenanthryl radical; alkaryl radicals, such as the o-, m- and p-tolyl radicals, xylyl radicals and ethylphenyl radicals.

Other examples are aralkyl radicals such as the benzyl radical and the α- and the ß-phenylethyl radical. Most preferred is the methyl radical.

According to a second aspect there is provided a laundry detergent composition for machine or hand washing comprising:

(i) an antifoam powder in an amount of 0.3 to 2.5 % by weight relating to the total amount of the laundry detergent composition, the antifoam powder comprising:

(a) 10 to 35 % by weight of the modified amino silicone antifoam fluid of formula (I) and

(b) 65 to 90 % by weight of a carrier filler selected from the group consisting of sodium carbonate, sodium sulphate, aluminium silicate, potassium carbonate, potassium sulphate, sodium bicarbonate, potassium bicarbonate and zeolite; and

(ii) usual laundry detergent ingredients.

DETAILED DESCRIPTION OF THE INVENTION

The term “comprising” is meant not to be limiting to any subsequently stated elements but rather to encompass non-specified elements of major or minor functional importance. In other words the listed steps, elements or options need not be exhaustive. Whenever the words “including” or “having” are used, these terms are meant to be equivalent to “comprising” as defined above.

Except in the operating and comparative examples, or where otherwise explicitly indicated, all numbers in this description indicating amounts of material ought to be understood as modified by the word “about”.

It should be noted that in specifying any range of concentration or amount, any particular upper concentration can be associated with any particular lower concentration or amount.

The terms weight percent, percent by weight, % by weight, wt%, and the like are synonyms that refer to the concentration of a substance as the weight of that substance divided by the weight of the composition and multiplied by 100.

The recitation of numerical ranges by endpoints includes all numbers subsumed within that range (e.g. 1 to 5 includes 1, 1.5, 2, 2.75, 3, 3.80, 4, and 5).

As used in this specification and the appended claims, the singular forms "a", "an", and "the" include plural referents unless the content clearly dictates otherwise. Thus, for example, reference to a composition containing "a compound" includes a mixture of two or more compounds. As used in this specification and the appended claims, the term "or" is generally employed in its sense including "and/or" unless the content clearly dictates otherwise.

For a more complete understanding of the above and other features and advantages of the invention, reference should be made to the following detailed description of preferred embodiments.

As discussed hereinbefore, in one aspect, the present invention relates to laundry detergent composition for machine or hand wash comprising an antifoam powder involving delayed-release antifoam.

We have found that preferred detergent compositions produce lower foam during the rinsing cycle and practically nil foam after two rinses. On the other hand, the preferred compositions produce high levels of foam during the washing cycle. These two opposing requirements are met by compositions of the invention.

Preferred detergent compositions include:

Surfactants

The detergent compositions include 2 wt% to 70 wt % surfactant, most preferably 10 to 30 wt% surfactants. Preferred compositions include anionic or non-ionic surfactants. More preferred compositions include a mixture of an anionic surfactant and a non-ionic surfactant.

In general, the non-ionic and anionic surfactants may be chosen from the surfactants described in "Surface Active Agents" Vol. 1, by Schwartz & Perry, Interscience 1949, Vol. 2 by Schwartz, Perry & Berch, Interscience 1958, in the current edition of "McCutcheon's Emulsifiers and Detergents" published by Manufacturing Confectioners Company or in "Tenside-Taschenbuch", H. Stache, 2nd Edn., Carl Hauser Verlag, 1981. Preferably the surfactants are saturated.

Suitable anionic surfactants which may be used are usually water-soluble alkali metal salts of organic sulphates and sulphonates having alkyl radicals containing from about 8 to about 22 carbon atoms, the term alkyl being used to include the alkyl portion of higher acyl radicals. Examples of suitable synthetic anionic detergent compounds are sodium and potassium alkyl sulphates, especially those obtained by sulphating higher C_5 to C_{15} alcohols, produced for example from tallow or coconut oil, sodium and potassium alkyl C_8 to C_{20} benzene sulphonates, particularly sodium linear secondary alkyl C_{10} to C_{15} benzene sulphonates; and

sodium alkyl glyceryl ether sulphates, especially those ethers of the higher alcohols derived from tallow or coconut oil and synthetic alcohols derived from petroleum.

The highly preferred anionic surfactants are sodium C_6 to C_{15} alkyl benzene sulphonates (commonly called LAS) and sodium C_{12} to C_{15} alkyl sulphates.

Suitable nonionic surfactants which may be used include, in particular, the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide (EO) either alone or with propylene oxide. Specific nonionic detergent compounds are C_6 to C_{22} alkyl phenol-ethylene oxide condensates, generally 5 to 25 EO, i.e. 5 to 25 units of ethylene oxide per molecule, and the condensation products of aliphatic C6 to C18 primary or secondary linear or branched alcohols with ethylene oxide, generally 5 to 40 EO.

It is preferred that a mixture of anionic and nonionic surfactants, in particular the groups and examples of anionic and nonionic surfactants pointed out in EP-A-346 995 (Unilever) is used.

Preferred compositions may include 1 wt% to 25 wt% nonionic surfactants.

In addition to these surfactants, other known surfactants such as cationic surfactants, amphoteric surfactant and zwitterionic surfactants may also be present in preferred compositions.

Preferred compositions include foaming surfactants, e.g. SLES (Sodium lauryl ether sulphate) or PAS (Primary alcohol sulphates) or a mixture thereof. Such compositions provides an ideal balance between preferred initial (high) lather and preferred final (very low) lather levels.

In detergent powders, generally the main foaming component is the anionic surfactant. When a preferred detergent composition containing powdered form of the silicone antifoam fluid is dissolved in water; large amount of foam gets generated. Without wishing to be bound by theory it is believed that at this stage, relatively little, or perhaps no interaction takes place between the amino groups of the modified silicone fluid and the anionic surfactants, presumably because of the presence of higher foam in the liquor.

As soon as soiled fabrics come in contact with the liquor during the washing cycle, the modified amino silicone fluid penetrates inside the fibers due to its high affinity to the fabrics. However, at this stage the quantity of lather is too high for the anionic surfactants to interact with the amino groups of the modified silicone so that any salt may be formed.

Therefore, the foaming nature of the detergent composition is not affected. During the rinsing cycle, the residual surfactants get mixed with fresh dose of water. It is believed that at this stage the foam density is comparatively lower. It is further believed that at this stage, the anionic surfactants get an opportunity to interact easily with the modified amino silicone which has penetrated and attached to the fabric, as the foam is much lower.

It is believed that a water-soluble silicone salt is formed, which mixes with the rinsing liquor. Therefore, the anionic surfactant loses its foaming nature. This leads to reduced foam during the rinsing cycle. We have observed that when preferred detergent compositions are used during domestic hand wash laundry method, practically no foam is observed after one or two rinses.

Therefore, preferred detergent compositions allow significant reduction in environmental impact by saving substantial quantity of rinse-water.

Further, we have also observed that fabrics washed with preferred detergent compositions were surprisingly found to be much softer to touch, which indicates good conditioning effects, presumably because of the residual silicone adhering onto the fabrics. Thus, according to another aspect the invention provides use of a laundry detergent composition of the first aspect for softness benefits on washed fabrics.

Suitable modified amino silicone antifoam fluids and powders thereof may be procured from Wacker Metroark Chemicals Pvt Ltd, India. Suitable silicone fluids and powders have been described in unpublished co-pending Indian Application 198/KOL/2010 filed by Wacker Metroark Chemicals Pvt Ltd, India.

Other optional ingredients

In addition to a surfactant and the modified amino silicone antifoam fluid, preferred compositions may include one or more of the following ingredients.

Builders or complexing agents

Builders are often included in detergent composition in order to reduce the concentration of free water hardness ions in the wash liquor. Ions such as Ca^{2+} and Mg^{2+} react with anionic surfactants, such as LAS, and cause precipitation.

Builders may be selected from calcium sequestrant materials, precipitating materials, calcium ion-exchange materials and mixtures thereof. Examples of calcium sequestrant builders include alkali metal polyphosphates, such as sodium tripolyphosphate (STPP) and organic sequestrants, such as ethylene diamine tetra-acetic acid. Examples of precipitating builders include sodium orthophosphate and sodium carbonate. Examples of calcium ion-exchange builders include the various types of water-insoluble crystalline or amorphous aluminosilicates, of which zeolites are the best known representatives, e.g. zeolite A, zeolite B (also known as zeolite P), zeolite C, zeolite X, zeolite Y and also the zeolite P-type. Low cost formulations may preferably include carbonate (including bicarbonate and sesquicarbonate) and/or citrates as builders. It is preferred to use carbonates as builders.

Where a builder is present, the compositions may suitably contain less than 20 wt %, preferably less than 10 wt% by weight, and most preferably less than 10 wt% builders. Preferred compositions include non-phosphate builder. Non-phosphate builders are preferred as they are environment-friendly.

Shading dyes

As used herein the term “shading dye” means dyes which when formulated in detergent compositions can deposit onto fabrics when the fabrics are contacted with wash liquor having the detergent compositions, thus altering the tint of the fabric through absorption of visible light. Shading dyes are also known as hueing agents. Preferred compositions include at least one shading dye.

Shading dyes deposit onto fabrics during the wash or rinse step, providing a visible hue to the fabric.

Shading of white fabrics may be done with any colour depending on consumer preference. Blue and violet are particularly preferred shades and consequently preferred dyes or mixtures of dyes are ones that give a blue or violet shade on white fabrics. Therefore preferred shading dyes are blue or violet.

Such dyes give a blue or violet colour to white fabrics. The preferred hue angle is 240° to 345°, more preferably 260° to 320° and most preferably 270° to 300°.

Shading dyes may be classified into several classes and in several ways. One way is to classify the dyes depends on their structures. Examples include azo dyes and anthraquinone dyes. Another way is to classify them according to their mode of application. Examples include direct dyes and acid dyes, disperse, vat, and solvent dyes. According to another method of classification, dyes are called hydrophobic or hydrophilic depending on their affinity for fabrics.

Yet another way of classifying shading dyes depends on whether the dyes deposit onto fabrics after a single-wash to show their effect, or whether they deposit after multiple washes. Dyes that deposit in a single-wash are called one-wash dyes.

Examples include Acid Violet 50 (AV50). The others are called build-up dyes. Some examples include Direct Violet 9 (DV9) and Solvent Violet 13 (SV13). Other preferred dyes may be selected from the chemical classes of benzodifuranes, methine, triphenylmethanes, napthalimides, pyrazole, phthalocyanine napthoquinone, anthraquinone and mono-azo or di-azo dyes.

The dye may also be a disperse dye such as Disperse Violet 27 (DV27), Disperse Violet 26 (DV26), Disperse Violet 28 (DV28), Disperse Violet 63 (DV63) and Disperse Violet 77 (DV77). Disperse Violet 28 (DV28) is the most preferred disperse dye. Particularly preferred hydrophobic dyes are SV13 and DV28; and DV28 is the most preferred hydrophobic dye. Preferred compositions include 0.0001 wt % to 0.008 wt%, more preferably 0.0003 wt% to 0.006 wt% hydrophobic dye. When the hydrophobic dye is DV28, the preferred range is 0.001 wt% to 0.006 wt%. When the hydrophobic dye is SV13, the preferred range is 0.0003 wt% to 0.0025 wt%. It is preferred that DV28 is included in the form of an adjunct.

The adjunct may preferably be made of inorganic carriers like soda ash, Sodium sulphate or zeolite. The adjunct may also include a dispersant e.g. lignin sulphonate. The dye may also

be a Direct dye. Non-limiting examples of these dyes are Direct Violet (DV) 5, 7, 9, 11, 26, 31, 35, 41 and 51 and DV99.
Further non-limiting examples of these dyes are also Direct Blue 34, 70, 71, 72, 75, 78, 82, and 120.

The most preferred direct dye is Direct Violet 9 (DV9). DV99 is also preferred. Such dyes have been described in WO2005/003274 A1 (Unilever). DV9 may be sourced from BASF.

Fluorescent agents

In order to further improve whiteness, preferred compositions may include a fluorescent agent (also called optical brightener). Fluorescent agents are well known and many such fluorescent agents are available commercially. Usually, these fluorescent agents are supplied and used in the form of their alkali metal salts, for example, the Sodium salts. Total amount of the fluorescent agent or agents which may be used in preferred compositions is generally from 0.005 wt% to 2 wt %, more preferably 0.01 wt% to 0.1 wt %. Preferred classes of fluorescer include di-styryl biphenyl compounds, e.g. TINOPAL® CBS-X, Di-amine stilbene di-sulphonic acid compounds, e.g. TINOPAL® DMS pure Xtra and BLANKOPHOR® HRH, and Pyrazoline compounds, e.g. BLANKOPHOR® SN. Preferred fluorescers are: sodium 2 (4-styryl-3-sulfophenyl) -2H-napthol [1, 2-d] trazole, disodium 4,4'- bis { [(4-anilino-6- (N methyl-N-2 hydroxyethyl) amino 1,3,5- triazin-2-yl)] amino } stilbene-2-2 ' disulfonate, disodium 4, 4 ' -bis { [(4-anilino-6-morpholino-l, 3,5-triazin-2-yl)] amino } stilbene-2-2' disulfonate, and disodium 4, 4 '-bis (2- sulfoslyryl) biphenyl .

Bleach

The function of a bleach is the discolouration and the removal of coloured stains such tea, wine, fruit and also of some clay types. Chromophores are disrupted and stains made more polar so that they are better removed. Another function of the bleach system is to kill bacteria.

The oxygen bleach system as is now being used for almost all of the modern bleach containing laundry detergents consists of TAED (tetra acetyl ethylene diamine) and a solid source of hydrogen peroxide (H_2O_2). This combination generates peracetic acid in combination with hydrogen peroxide.

The bleach may also be of the chlorine type, such as hypohalites, e.g. Calcium hypochlorite. Reductive bleaches, such as Sodium meta bi sulphite ($Na_2S_2O_5$), Sodium Sulphite (Na_2SO_3), perborates or borohydrides may also be used.

Percarbonates, such as Sodium percarbonate (Na_2CO_3.1.5 H_2O_2 are the most preferred bleaches. Commercially available materials contain approxmately 13 to 14 %, generally around 13.25 % available Oxygen. Percarbonates have good low temperature solubility, storage stability, and they decomposes into carbonates which are environmentally more acceptable than borates.

Preferred compositions may also include 2 to 25 wt%, more preferably 10 to 22 wt%, and most preferably 12 to 22 wt% percarbonate.

Enzymes

Preferred compositions may also include one or more enzymes, which are usually included to counteract stains. Enzymes are known to be substrate-specific in their action, so it is very common to find detergent compositions with a combination of enzymes.

Lipase (also known as esterase) is an enzyme which catalyses hydrolysis of ester bonds of edible fats and oils, i.e. triglycerides, into free fatty acids, mono- and diglycerides and glycerol. It is believed that the primary function of lipase is to reduce build-up of sebum. Lipase is also suitable for detergent compositions that contain higher amount of anionic surfactants, typically 20 to 40 wt%. Lipase is also believed to remove difficult stains like tomato oil, pasta sauce, pesto, motor oil, colourless oils like olive oil and corn oil. Preferred lipase enzymes include those of bacterial or fungal origin.

Chemically modified or protein engineered mutants may also be used. Preferred lipase enzymes are available under the trademarks LIPOCLEAN®, LIPOLASE®, LIPOLASE® Ultra and LIPEX®. LIPEX® is particularly preferred, and LIPEX® 100 TB is further particularly preferred. The activity of commercial lipase is commonly expressed as Lipase Units or LU. Different lipase preparations may have different activities. For fungal lipases these may range from 2,000 to 2,000,000 LU per gram. The activity may also be represented as FIP units/g or FCC III LU/g. One of these new Lipase Units is equivalent to ten of the old LU, or 1,000 FIP units/g = 10,000 LU/g.

Preferred compositions may include lipase having 5 to 20000 LU/g.

In addition to lipase, one or more other enzymes may also be present in preferred compositions. Such enzymes include proteases, alpha-amylases, cellulases, peroxidases/oxidases, pectate lyases, and mannanases.

Suitable proteases include those of animal, vegetable or microbial origin. Microbial origin is preferred. Chemically modified or protein engineered mutants are included.

The protease may be a serine protease or a metallo-protease, preferably an alkaline microbial protease or a trypsin-like protease. Preferred commercially available protease enzymes include ALCALASE®, SAVINASE®, PRIMASE®, DURALASE ®, DYRAZYM®, ESPERASE®, EVERLASE®, POLARZYME®, KANNASE®, MAXATASE®, MAXACAL®, MAXAPEM®, PROPERASE®, PURAFECT® and PURAFECT® OxP.

Suitable amylases (alpha and/or beta) include those of bacterial or fungal origin. Chemically modified or protein engineered mutants are included.
Amylases include, for example, alpha-amylases obtained from Bacillus, e.g. a special strain of *B. lichenformis*.

Commercially available amylases are DURAMYL®, TERMAMYL®, TERMAMYL® Ultra, NATALASE®, STAINZYME®, FUNGAMYL® BAN®, RAPIDASE® and PURASTAR®.

Suitable cellulases include those of bacterial or fungal origin. Chemically modified or protein engineered mutants may also be used. Suitable cellulases include cellulases from the genera *Bacillus, Pseudomonas, Humicola, Fusarium, Thielavia, Acremonium*, e.g. the fungal cellulases produced from *Humicola insolens, Thielavia terrestris, Myceliophthora thermophila , and Fusarium oxysporum*. Commercially available cellulases include CELLUZYME®, CAREZYME®, ENDOLASE®, RENOZYME®, CLAZINASE® and PURADAX® HA. Suitable peroxidases/oxidases include those of plant, bacterial or fungal origin. Chemically modified or protein engineered mutants may also be used. Examples of useful peroxidases include peroxidases from Coprinus, e.g. from *C. cinereus*, and variants thereof as those described in WO 93/24618, WO 95/10602, and WO 98/15257. Commercially available peroxidases include GUARDZYME® and NOVOZYM® 51004.

Enzyme stabilizer

When enzymes are present, it is common to include a stabilizer. Any enzyme present in the composition may be stabilized using conventional stabilizing agents, e.g., a polyol such as propylene glycol or glycerol, a sugar or sugar alcohol, lactic acid, boric acid, or a boric acid derivative, e.g., an aromatic
borate ester, or a phenyl boronic acid derivative such as 4-formylphenyl boronic acid, and the composition may be formulated as described in e.g. WO 92/19709 and WO 92/19708.

Metal chelating agents

The compositions may include a metal chelating agent such as carbonates, bicarbonates, and sesquicarbonates. The metal chelating agent can be a bleach stabiliser (i.e. heavy metal sequestrant).

Suitable metal chelation agents include ethylenediamine tetraacetate (EDTA), diethylenetriamine pentaacetate (DTPA), ethylenediamine disuccinate (EDDS), and the polyphosphonates such as the DEQUESTS®, ethylenediamine tetramethylene phosphonate (EDTMP) and diethylenetriamine pentamethylene phosphate (DETPMP).

Polymers

The compositions may include one or more polymers for soil-release and anti-redeposition of dirt. Anti-redeposition agents are incorporated to reduce the redeposition of soil which was removed from the load during the wash cycle.
Soil release agents improve the removal of soil from a fabric on which a film of such an agent was deposited in the previous wash(es) Examples are carboxymethylcellulose, poly (vinylpyrrolidone) , poly(ethylene glycol), poly(vinyl alcohol), poly(vinylpyridine- N-oxide) , poly(vinylimidazole) , polycarboxylates such as polyacrylates, maleic/acrylic acid copolymers and lauryl methacrylate/acrylic acid copolymers.

Modern detergent compositions typically employ polymers as dye-transfer inhibitors (DTI). These prevent migration of dyes, especially during long soak times. Any suitable dye-transfer inhibitor may be used in preferred compositions. Generally, such dye-transfer inhibitors

include polyvinyl pyrrolidone polymers, polyamine N-oxide polymers, copolymers of N-vinylpyrrolidone and N-vinylimidazole, manganese pthalocyanine, peroxidases, and mixtures thereof.

Nitrogen-containing, dye binding DTI polymers are preferred. Of these polymers, co-polymers of cyclic amines such as vinyl pyrrolidone, and/or vinyl imidazole are preferred.

Copolymers of N-vinylpyrrolidone and N-vinylimidazole polymers (as a class, referred to as "PVPVI") are also preferred. These copolymers can be either linear or branched. Suitable PVPVI polymers include SOKALAN® HP56, available commercially from BASF.

Perfume

Preferred compositions may also include perfumes. The perfumes could be of natural origin or synthetic. They include single compounds and mixtures. Specific examples of such components may be found in Perfume and Flavor Chemicals by S. Arctander 1969, Montclair, N.J. (USA). By perfume in this context is not only meant a fully formulated product fragrance, but also selected components of that fragrance, particularly those which are prone to loss, such as the so-called top notes. The perfume may be used in the form of neat oil or an encapsulated form.

Other well-known ingredients include salts like Sodium chloride and Sodium sulphate, flow aids such as Calcite and Dolomite, bleaches, such as peroxy bleach compounds or percarbonates, bleach stabilisers such as phosphonates, bleach activators e.g. tetra-acetyl ethylenediamine (TAED), Sodium silicate; coloured speckles; visual cues and fabric conditioning compounds. These may be included during the process of manufacture, if the ingredients are sufficiently robust. Alternatively the ingredients may be post-dosed in the detergent composition, as is well known to those skilled in the art.

The detergent composition may be made by spray drying process or by a non-tower (NTR) process. Accordingly the detergent compositions may be fully spray dried or fully non-tower powders. A mixture of spray dried powder and non-tower powder is preferred. Particularly preferred compositions include 50 to 90 parts non-tower (NTR) detergent powder and 50 to 10 parts spray dried detergent powder. Most preferred combination is 70 parts NTR powder

and 30 parts spray-dried powder. Preferably, a powdered form of the modified amino silicone fluid is post-dosed to the powder.

Method

According to another aspect the invention provides a method of washing fabrics by hand or in a machine comprising:

(i) a step of contacting the fabrics with an aqueous wash liquor of a composition of the first aspect; and,

(ii) a step of rising the fabrics, wherein the method requires substantially lower amount of water for rinsing.

Preferred detergent compositions need only 1 or 2 rinses; as compared to prior art detergent compositions that typically require 4 to 5 rinses.

The details of the invention, its nature and objects are explained hereunder in greater detail in relation to the following non-limiting examples.

EXAMPLES

Example-1: A preferred detergent composition (Composition-1) containing a powder of a modified amino silicone antifoam fluid

Formulation of (Composition-1) was as follows:

Linear Alkyl benzene sulfonate Salt	=	14.00 parts
Sodium tripolyphosphate	=	26.40 parts
Soda Ash	=	47.05 parts
Sodium Sulphate	=	4.40 parts
Sodium Perborate	=	6.85 parts
Tetra Acetyl Ethylene Diamine	=	0.5 parts
Perfume	=	0.30 parts
Antifoam powder*	=	0.5 parts

* The antifoam powder was made as described in co-pending Indian application 198/KOL/2010.

Comparative Example-1: Detergent composition without antifoam powder (Comparative Composition-1)

Formulation of the comparative detergent composition was as follows:

Linear Alkyl benzene sulfonate Salt	=	10.00 parts
Soap	=	1.25 parts
Lauryl alcohol 7 EO	=	2.00 parts
Sodium tripolyphosphate	=	26.40 parts
Soda Ash	=	47.9 parts
Sodium Sulphate	=	4.40 parts
Enzyme Protease	=	0.40 parts
Sodium Perborate	=	6.85 parts
Tetra Acetyl Ethylene Diamine	=	0.5 parts
Perfume	=	0.30parts

Example-2: A preferred detergent composition (Composition-2) containing a powder of a modified amino silicone antifoam fluid

Formulation of Composition-2 was as follows:

Linear Alkyl benzene sulfonate Salt	=	10.00 parts
Soap	=	1.25 parts
Lauryl alcohol 7 EO	=	2.00 parts
Sodium tripolyphosphate	=	26.40 parts
Soda Ash	=	47.40 parts
Sodium Sulphate	=	4.40 parts
Enzyme Protease	=	0.40 parts
Sodium Perborate	=	6.85 parts
Tetra Acetyl Ethylene Diamine	=	0.5 parts
Perfume	=	0.30 parts
Antifoam powder*	=	0.5 parts

* The antifoam powder was made as described in co-pending Indian application 198/KOL/2010.

Performance evaluation (hand wash)

Performance of the preferred and the comparative detergent compositions was compared by following a bucket-wash protocol. The steps and requirements will now be described.

(i) 24 °FH hard water;

(ii) fabric to liquor ratio was 1:10;

(iii) powder dosage 4 gpl detergent (gpl = grams per liter);

(iv) the water was whisked for 20 seconds and the fabrics were immersed in the bucket;

(v) the washing cycle was maintained for 15 to 30 minutes and the foam height was measured at every 5 to 10 minute intervals;

(vi) the fabrics were squeezed to 50 % weight pick up;

(vii) the fabrics were rinsed with fresh supply of 24 °FH hard water at a ratio of 1 part rinse water to 7 parts of fresh water; and,

(viii) the fabrics were squeezed again to 50 % weight pick up. The foam height was measured again.

(ix) The steps were repeated to get the foam height after the second, third and fourth rinse.

Performance evaluation (Machine wash)

Performance of the preferred and the comparative detergent compositions was compared in a top load automatic washing machine. The steps and requirements will now be described. 24 °FH hard water;

(i) fabric to liquor ratio was 1:10;

(ii) detergent powder dosage 4 gpl detergent (gpl = grams per liter);

(iii) washing cycle was maintained for 15 to 30 minutes. The top lid of washing machine was opened just before the machine was to discharge the washing liquor. The fabrics were taken out and squeezed so that fabric retained 50% wash liquor. Thereafter, the foam height was measured.

(iv) The top lid was closed and the machine was allowed to run again. As soon as machine discharged the washing liquor, the top lid was opened and the fabrics were transferred to the machine. The top lid was closed and the machine was in operation for the rinsing cycle. The top lid was opened just before the first rinsing liquor was to be discharged. The fabrics were taken out and squeezed so that only 50% wash liquor remained. The foam height was measured again and it was noted as foam height after first rinse.

(v) The steps were repeated to get the foam height after the second, third and fourth rinse.

Results of foam-height are shown in tables 1 and 2.

Table –1: Hand washing result

Sample's name	detergent dosage, gpl	washing time, minutes	foam height during washing, cm				Foam height after 1st rinse, cm	Foam height after 2nd rinse, cm	Foam height after 3rd rinse, cm	Foam height after 4th rinse, cm
			0 min	10 min	20 min	30 min				
Comparative Comp'n-1	4	30	10.5	10.5	10	9	2	1.5	1	0.5
Comp'n-1	4	30	12	10	10	9	0.5	0	-	-

Table 2: Machine Wash result

Sample's name	detergent dosage, gpl	washing time, minutes	foam height after 30 minutes washing/ in cm	Foam height after 1st rinse, cm	Foam height after 2nd rinse, cm	Foam height after 3rd rinse, cm	Foam height after 4th rinse, cm
Comparative Comp'n-1	4	30	11	2	1.6	1.0	0.8
Comp'n-2	4	30	11.5	0.8	0.2	0	-

The data in tables 1 and 2 indicate that preferred detergent compositions performed better than conventional detergent composition. Preferred detergent compositions showed higher foam height during the wash cycle, but the foam height during rinsing step was much lower, and practically nil after the second rinsing step. On the other hand, comparative detergent composition showed higher foam height during the wash cycle, as well as during each of the rinsing steps.

Example-3

A preferred detergent powder (Composition-3) was made, which was a 70:30 mixture of a NTR (non-tower route) component and a spray dried component. The formulation contained foaming surfactants SLES (Sodium lauryl ether sulphate) and PAS (Primary alcohol sulphate) along with a powder of a preferred modified amino silicone antifoam fluid described in co-

pending application 198/KOL/2010 filed by Wacker Metroark Chemicals Pvt Ltd. This formulation mix provides an ideal balance between initial lather and final lather levels.

Table-3

Ingredient	Wt%
LAS	19
Soda ash (light)	30
zeolite	8
Sodium silicate	2
Sodium Sulphate	5
Sodium Chloride	7
dolomite	4
TINOPAL® CBSX	0.8
NDOM, moisture, Inorganic ingredients	4
savinase	0.1
perfume	0.5
Antifoam powder*	1
AV-50 granules (shading dye)	1
SLES granules	3
Total including other minors	100

Results of an in-house consumer panel test with 300 consumers indicated that this preferred composition was rated significantly better on several attributes e.g. “reduces effort and time during rinse”. Its performance was compared against commercially available ARIEL® detergent powder and another control composition (Control Composition-2). This Control composition-2 had every ingredient in table-3 except the antifoam powder.

Table-4 describes the comparative data between preferred composition (Composition-3) and ARIEL® detergent powder.

Table-4

Attributes	Composition 3	ARIEL®
overall opinion on the product	5.82	5.81
cleans clothes thoroughly	4.86	4.86
overall opinion of fragrance	5.66	5.72b
requires less water at rinse stage	4.63	4.63
reduces effort and time at rinsing stage	4.62	4.59
requires less number of rinses	4.6	4.58

In addition to the benefits described in table-4, we found that fabrics washed with the preferred composition (Composition-3) had a lingering perfume impact. Accordingly in another aspect the invention provides use of a composition of the first aspect for lingering perfume impact on washed fabrics. We also found that wash liquor of the preferred composition (Composition-3) had comparatively higher perfume impact. The wash liquor was also found to be highly soapy by the consumers when compared to the results with ARIEL® detergent powder. Such effects, which are presented in table-5, were not seen with the control composition-2.

Table-5

Attributes	Control Comp'n-2	Comp'n-3	LSD	p value	Significance
perfume impact while dissolving (less to more)	56.5	64.8A	4.91	0.001	***
un-dissolved particles (less to more)	56.8	59.4	2.73	0.058	*
soapiness in solution (less to more)	54.2	61.7A	5.32	0.007	***
speed of lather (fast to slow)	43.5B	48.1	3.37	0.009	***
amount of lather (less to more)	103.8	99.9	4.26	0.074	*
soapiness on pillow cover (less to more)	57.3	61.5	4.29	0.055	*
amount of lather (to generate lather) (less to more)	95.4	91.4	4.33	0.069	*
amount of lather (from rinsing) (rinse 1) (less to more)	53.0B	45.7	4.93	0.005	***
amount of lather (from rinsing) (rinse 2) (less to more)	32.3B	26.3	3.73	0.002	***
amount of lather (from rinsing) (rinse 3)	18.4B	14.2	2.18	0.000	****
amount of lather (from rinsing) (rinse 4) (less to more)	10.8B	8.6	1.44	0.004	***
transparency of solution (rinse 3) (less to more)	117.8	121.8A	3.67	0.037	**
transparency of solution (rinse 4) (less to more)	138.9	141.4A	1.49	0.002	***

Note: higher number of asterisks (*) associated with “significance” means the observed values were highly significant. For example, a value with 3 asterisks is statistically more significant then a comparative value with 2 asterisks.

The data in table-5 indicates that the preferred composition (Composition-3) showed comparatively higher perfume impact, higher soapiness, higher speed of lather, comparable amount of lather while dissolving, and comparatively lower lather at every rinse stage. This was further corroborated by the higher transparency of rinse liquor at every rinse stage.

It will be appreciated that the illustrated examples provide detergent compositions containing silicone antifoams which show improved delayed-release antifoam action.

It should be understood that the specific forms of the invention herein illustrated and described are intended to be representative only as certain changes may be made therein without departing from the clear teachings of the disclosure.

Although the invention has been described with reference to specific embodiments, it will be appreciated by those skilled in the art that the invention may be embodied in many other forms.

Claims

1. Laundry detergent composition for machine or hand washing comprising :

(i) a modified amino silicone antifoam fluid of the formula:

$\mathbf{X}R_2Si(OSi\mathbf{A}R)_n(OSiR_2)_mOSiR_2\mathbf{X}$ (I)

where

A is an amino radical of the formula

$-R^1-[NR^2-R^3-]_xNR^2_2$

X is **R** or a polyoxyalkylen group **G** of the formula

$-R^4-(O-R^5)_y-O-R^6$

R is a monovalent hydrocarbon radical having from 1 to 18 carbon atoms,

$\mathbf{R^1}$ is a divalent C_1-C_{10}-alkylen radical, preferably a radical of the formula $-CH_2CH_2CH_2-$,

$\mathbf{R^2}$ is a hydrogen atom or a C_1-C_4-alkyl radical, preferably a hydrogen atom,

$\mathbf{R^3}$ is a divalent C_1-C_{10}-alkylen radical, preferably a radical of the formula $-CH_2CH_2-$,

$\mathbf{R^4}$ is a divalent C_1-C_{10}-alkylen, preferably a radical of the formula $-CH_2CH_2CH_2-$,

$\mathbf{R^5}$ is a C_1-C_4-alkylen radical, preferably a radical of the formula $-CH_2CH_2-$ or $-CH_2CH_2(CH_3)-$ or mixtures of them,

$\mathbf{R^6}$ is a hydrogen atom or an C_1-C_4-alkyl radical, preferably a hydrogen atom or a methyl radical, more preferably a hydrogen atom,

n is an integer from 1 to 6, preferably from 1 to 3,

m is an integer from 1 to 200, preferably from 1 to 80,

x is 0 or 1 and

y is an integer from 5 to 20, preferably from 5 to 12, with the provision that on average from 30 to 60 mol%, preferably 50 mol%, of radicals **X** are polyoxyalkylen groups **G**; and,

(ii) usual laundry detergent ingredients.

2. Laundry detergent composition as claimed in claim 1 wherein the amino radical (A) is selected from $-(CH_2)_3NH_2$;$-(CH_2)_3NH(CH_2)_2NH_2$;$-(CH_2)_3NHC_6H_{11}$;$-(CH_2)_3NH(CH_2)_2NHC_6H_{11}$; $-(CH(CH_3)CH_2CH_2)NH_2$;$-(CH(CH_3)CH_2CH_2)NH(CH_2)NH_2$; $-(CH_2)NH_2$;–

$(CH_2)NH\ (CH_2)_2\ NH_2$; $-(CH(CH_3)CH_2CH_2)NHC_6H_1$; $-(CH(CH_3)CH_2CH_2)NH(CH_2)NHC_6H_1$;$-(CH_2)NHC_6H_{11}$;$-(CH_2)NH\ (CH_2)_2\ NHC_6H_{11}$, its protonated amino forms and acylated amines of said amino radicals.

3. Laundry detergent composition as claimed in claim 1 or 2 wherein amine number of said modified silicone antifoam fluid is from 0.6 mg of KOH/g of polymer to 2.0 mg of KOH/ g of polymer of the silicone fluid.

4. Laundry detergent composition as claimed in any one of the preceding claims wherein in said formula (I) **X** involving polyoxyalkylen radical **G** is selected from $-(CH_2)_3-(OC_2H_4)_y-O-R^6$; $-(CH(CH_3)CH_2CH_2)-(OC_2H_4)_y-O-R^6$;$-(CH_2)_3-(OC_3H_6)_y-O-R^6$; $-(CH(CH_3)CH_2CH_2)-(OC_3H_6)_y-O-R^6$;$-(CH_2)-(OC_2H_4)_y-O-R^6$; $-CH_2-(OC_2H_4)_y-O-R^6$;$-(CH_2)-(OC_3H_6)_y-O-R^6$; or $-CH_2-(OC_3H_6)_y-O-R^6$.

5. A laundry detergent composition as claimed in any one of the preceding claims comprising:
   (i) an antifoam powder in an amount of 0.3 to 2.5 % by weight relating to the total amount of the laundry detergent composition, said antifoam powder comprising:
      (a) 10 to 35 % by weight of the modified amino silicone antifoam fluid of Formula (I); and,
      (b) 65 to 90 % by weight of a carrier filler selected from the group consisting of sodium carbonate, sodium sulphate, aluminium silicate, potassium carbonate, potassium sulphate, sodium bicarbonate, potassium bicarbonate and zeolite; and
   (ii) usual laundry detergent ingredients.

6. A laundry detergent composition as claimed in any one of the preceding claims comprising 2 wt% to 70 wt % surfactants.

7. A laundry detergent composition as claimed in claim 6 wherein said composition comprises a mixture of an anionic surfactant and a non-ionic surfactant.

8. A laundry detergent composition as claimed in claim 6 or 7 wherein said composition comprises Sodium lauryl ether sulphate or primary alcohol sulphates or a mixture thereof.

9. A laundry detergent composition as claimed in any one of the preceding compositions wherein said composition comprises a non-phosphate builder.

10. A laundry detergent composition as claimed in any one of the preceding claims wherein said composition comprises at least one shading dye.

11. A laundry detergent composition as claimed in any one of the preceding claims wherein said composition comprises 50 to 90 parts non-tower detergent powder and 50 to 10 parts spray dried detergent powder.

12. Use of a laundry detergent composition as claimed in claim 1 for softness benefits on washed fabrics.

13. Use of a composition as claimed in claim 1 for lingering perfume impact on washed fabrics.

14. A method of washing fabrics by hand or in a machine comprising;
    (i) a step of contacting said fabrics with an aqueous wash liquor of a composition as claimed in claim 1; and,
    (ii) a step of rising said fabrics, wherein said method requires substantially lower amount of water for rinsing.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2011/052775

A. CLASSIFICATION OF SUBJECT MATTER
INV. C11D3/37
ADD.

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)
C11D C08G

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)
EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	EP 0 685 250 A1 (DOW CORNING TORAY SILICONE [JP]) 6 December 1995 (1995-12-06) page 2, lines 1-4,28-53; claims 1-10; examples 1-4; table 1	1-14
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A	JP 11 158287 A (DOW CORNING TORAY SILICONE) 15 June 1999 (1999-06-15) the whole document	1-14

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

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"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

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"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

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
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INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2011/052775

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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INTERNATIONAL SEARCH REPORT

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