(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
25 May 2001 (25.05.2001)

PCT

(10) International Publication Number
WO 01/36580 A1

(51) International Patent Classification⁷: C11D 17/00, 3/39, 3/10, 17/06

(21) International Application Number: PCT/US00/30870

(22) International Filing Date: 10 November 2000 (10.11.2000)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
9926799.9 13 November 1999 (13.11.1999) GB

(71) Applicant *(for all designated States except US)*: **THE PROCTER & GAMBLE COMPANY** [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US).

(72) **Inventors; and**

(75) **Inventors/Applicants** *(for US only)*: **GOULD, Paul, Anthony** [GB/GB]; 6 Oaklands Drive, Hazel Grove, Stockport, Cheshire SK7 6LL (GB). **DAVIDSON, Nicola, Ethel** [GB/GB]; 15 The Wills Building, Wills Oval, The Coast Road, Newcastle upon Tyne NE7 7RW (GB). **SCHAMP, Susan, Judith** [GB/GB]; 31 Brook Street, Whitley Bay, Tyne & Wear NE26 1AF (GB). **SCHAMP, Koen, Mariette, Albert** [GB/BE]; 31 Brook Street, Whitley Bay, Tyne & Wear NE26 1AF (GB).

(74) **Agents: REED, T., David** et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217-1087 (US).

(81) **Designated States** *(national)*: AE, AG, AL, AM, AT, AT (utility model), AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, CZ (utility model), DE, DE (utility model), DK, DK (utility model), DM, DZ, EE, EE (utility model), ES, FI, FI (utility model), GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KR (utility model), KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SK (utility model), SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) **Designated States** *(regional)*: ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:
— *With international search report.*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 01/36580 A1

(54) Title: DETERGENT COMPOSITIONS

(57) **Abstract:** The present invention provides a detergent composition with a bulk density of less than 570g/1, characterised in that the percarbonate source comprises a salt of percarbonate, and that, after being exposed simultaneously to 32°C and 80% relative humidity for 24 hours, the detergent composition has a mean particle size of 100% to 130% of the original mean particle size. The present invention also provides a granular bleach-containing detergent composition with a bulk density of less than 570g/1 comprising a percarbonate source and less than 0.06% by weight extractable suds suppressing silicone, where the percarbonate source comprises a salt of percarbonate, characterized in that percarbonate source comprises less than 2% by weight chloride ions. The detergent composition is particularly applicable for use in climates comprising conditions of high temperature and high relative humidity.

Detergent Compositions

Technical Field

The present invention relates to bleach-containing detergents. This technology may find an application in laundry detergent compositions, soaking detergent compositions, or any other compositions for household applications or the like.

Background of the Invention

When detergent products are stored in a geographical location that has a hot and/or humid climate, an industrial environment, a household environment, or any combination thereof, where a high temperature and a high relative humidity are prevalent, it has been found that the mean particle size of the detergent composition increases during storage of the product. This increase in mean particle size, gives the product a coarse appearance of an aged product, which is not acceptable to the consumers, especially when the product is of a hand-wash application.

For many years, the laundry industry have been trying to determine the cause of this mean particle size increase of the detergent composition, with little success. However, the inventors have now successfully identified the cause of this mean particle size increase of the detergent composition during storage.

The laundry industry have been using perborate as a source of available oxygen in granular bleach-containing detergent compositions for use in hot or humid environments. The inclusion of a bleaching system in a granular detergent formulation helps to improve the cleaning performance of the detergent and is common practice in the laundry industry.

The inventors have found that in granular bleach-containing detergent compositions, it is the perborate that causes the increase in the mean particle size of the detergent composition during storage in conditions of high temperature and high relative humidity. Whilst not intending to be bound by theory, it is thought that in these conditions, sodium perborate monohydrate is hydrated to sodium perborate tetrahydrate. Sodium perborate tetrahydrate forms a gel that binds other detergent ingredients, thus forming detergent components of an irregular and increased size. This increase in the mean particle size of the detergent components results in a product that is of coarse appearance and not acceptable to the consumers.

The inventors have suprisingly found that when selected sodium percarbonate is used instead of sodium perborate as a source of available oxygen in a granular bleach-containing detergent composition with a bulk density of less than 570g/l, the problem of increasing particle sizes is alleviated. It has been found that when such products are stored under hot and/or humid conditions, for example, after being simultaneously exposed to 32°C and 80% relative humidity for 24 hours, the detergent composition retains its main particle size, in that it has a mean particle size of 100% to 130% of the original mean particle size. This results in a product that remains attractive to the consumer, which is of great benefit to the laundry industry and is especially applicable to handwash applications.

Also, the inventors have found that, after storage in such, the detergent composition retains its bulk density, in that it has a bulk density of 90% to 100% of the original bulk density. This results in a product that can still be effectively and accurately dosed during the washing process.

By using specifically selected sodium percarbonate in detergent compositions, namely sodium percarbonate which comprises less than 2% by weight chloride ions or sodium percarbonate prepared by a fluidised bed process, the inventors have found that detergent compositions comprising said sodium percarbonate are particularly stable.

Summary of the Invention

Summary of the Invention

The invention provides a granular bleach-containing detergent composition with a bulk density of less than 570g/l, characterised in that the detergent composition comprises a percarbonate source, and that, after being openly exposed simultaneously to 32°C and 80% relative humidity for 24 hours, the detergent composition has a mean particle size of 100% to 130% of the original mean particle size.

The invention also provides a granular bleach-containing detergent composition with a bulk density of less than 570g/l comprising a percarbonate source and less than 0.06% by weight extractable suds suppressing silicone, where the percarbonate source comprises a salt of percarbonate, characterised in that percarbonate source comprises less than 2% by weight chloride ions.

The invention is applicable to detergent compositions that are stored in a climate comprising conditions of high temperature and high relative moisture content.

Detailed Description of the Invention

Detergent Composition

The granular bleach-containing detergent composition has a bulk density of less than 570g/l, preferably from 200g/l to 550g/l, more preferably from 300g/l to 550g/l, even more preferably from 400g/l to 500g/l.

The granular bleach-containing detergent composition comprises a percarbonate source that comprises a salt of percarbonate, preferably sodium percarbonate, although in addition other forms of percarbonate can also be comprised by the percarbonate source of the invention. Also, in addition to percarbonate, other sources of available oxygen known in the art, or a combination thereof, can be included in the granular detergent composition, although it is preferred that percarbonate is the only perhydrate component included in the granular detergent composition.

Mean particle size

The mean particle size of the detergent composition is the mean particle size of all of the particles that the detergent composition comprises.

The granular detergent composition comprises a percarbonate source and optionally additional detergent ingredients. When the granular detergent composition is exposed to conditions of 32°C and 80% relative humidity for 24 hours, after exposure to these conditions, the detergent composition has a mean particle size of 100% to 130%, preferably 100% to 120%, more preferably 100% to 110% of the original mean particle size.

For the purpose of this invention, this is determined by the following:

100g of detergent composition comprising 20% by weight alkyl sulphate surfactant in a blown powder, less than 2% by weight other cationic or anionic, surfactants in a blown powder, 25% by weight sodium tripolyphosphate surfactants in a blown powder, 6% by weight 1.6 ratio silicate surfactants in a blown powder, 15% by weight carbonate surfactants in a blown powder, and 10% by weight sodium percarbonate (for example, see examples, sodium percarbonate sample 1 and 2) was placed, on a petri dish with no external barrier, in an incubator with controlled ambient conditions of 32°C and 80% relative humidity. The detergent composition was exposed to these said conditions in the incubator for 24 hours.

The mean particle size is determined before and after the detergent composition was exposed to the said conditions. The mean particle size of the detergent composition is measured using the method ISO 3118 (1976), where 100g of product is used to determine the mean particle size, the sieve sizes that are used are 250, 425, 600, 850, 1180 and 2000 micrometers, and the time the product is allowed in the sieves before the mean particle size is measured is 5 minutes. See example 1.

Bulk density

The granular detergent composition comprises a blown powder, a percarbonate source, and additional detergent ingredients, and has a bulk density less than 570g/l. When the detergent composition is stored in conditions of 32°C and 80% relative humidity for 8 weeks, after storage in these conditions, the detergent composition has a bulk density of 90% to 100% of the original bulk density.

For the purpose of this invention, this is determined by the following

1.5kg detergent composition comprising 20% by weight alkyl sulphate surfactant in a blown powder, less than 2% by weight other cationic or anionic surfactants in a blown powder, 25% by weight sodium tripolyphosphate in a blown powder, 6% by weight 1.6 ratio silicate in a blown powder, 15% by weight carbonate in a blown powder, and 10% by weight sodium percarbonate (for example, see examples, sodium percarbonate example 1 and 2) was stored, in packaging typical of laundry detergent products, in a room with controlled ambient conditions of 32°C and 80% relative humidity. The detergent composition was stored in these said conditions for 8 weeks.

The bulk density is determined before and after the detergent composition was stored in these said conditions. The bulk density of the detergent composition is measured using the method ISO 3424 (1975), see example 2.

Other preferred components of the detergent composition

The detergent composition can comprise an alkali or earth metal salt of phosphate. The alkali or earth metal salt of phosphate can be any known in the art, or combination thereof. Preferably this is sodium phosphate or sodium tripolyphosphate. The detergent composition comprises less than 36%, preferably from 1 to 35%, more preferably from

10% to 35%, even more preferably from 12% to 30%, by weight an alkali or earth metal salt of phosphate. Detergent compositions containing a low level of, or no, phosphate can also be used by this invention. Typical sources of phosphate that can be comprised by the composition of the invention are described hereinafter.

The detergent composition is preferably a high sudsing detergent formulation. These can be any high sudsing detergent formulations known in the art, or part thereof. The detergent composition can comprise less than 5%, preferably less than 2%, more preferably less than 1% by weight a suds suppresser. The suds suppresser can be any known in the art, or combination thereof. Preferably the suds suppresser is either a soap, paraffin, wax, or any combination thereof. Also, the detergent composition can comprise less than 0.06% by weight extractable suds suppressing silicone. The extractable suds suppressing silicone can be any known in the art, or any combination thereof. Typical suds suppressers, including suds suppressing silicone are described hereinafter.

The detergent composition has a moisture content of less than 10%, preferably 6% to 9% by weight.

When used herein, 'moisture content' means for the purpose of the invention, any free water molecules present in the detergent composition or water molecules in the form of hydrates, such as present in sodium phosphate, usually this is sodium tripolyphosphate.

The reserve alkalinity gives a measure of the buffering capacity of the detergent. This is the amount of NaOH or detergent composition required to neutralise 0.2M hydrochloric acid to pH 7.5. The granular detergent composition has a preferred reserve alkalinity of at least 6g NaOH per 100g detergent composition, measured to a pH of 7.5.

The detergent composition comprises 0.05% to 2%, preferably 0.1% to 2%, more preferably 0.2% to 1.5%, even more preferably 0.5% to 1.0%, by weight a metal chelating agent. The metal chelating agent can be any agent known in the art, or combination thereof, that chelates heavy metal ions, as described hereinafter.

Bleaching System

The granular detergent composition of the invention comprises a source of percarbonate. The percarbonate source acts as a source of available oxygen. Other sources of available oxygen can also be comprised by the detergent composition of the invention, to form a bleaching system. Typical bleaching systems comprised by the detergent composition are described hereinafter.

When used herein, 'bleaching system' means for the purpose of the invention any bleaching system known in the art that comprises the percarbonate source and an additional bleaching compound, preferably a peracid or precursor thereto.

Percarbonate Source

When used herein, 'percarbonate source' means for the purpose of the invention, any source of percarbonate, typically comprising a salt of percarbonate, preferably the salt of percarbonate is sodium percarbonate.

The detergent composition comprises a percarbonate source at a level of from 0.01% to 12%, preferably from 2% to 10%, more preferably from 3% to 7%, by weight of the detergent composition.

The percarbonate source of the invention can be prepared by any method known in the art, preferably a fluidised bed process. The fluidised bed process can be any known in the art.

The percarbonate source preferably comprises less than 2%, preferably less than 1.5%, more preferably less than 1.2%, even more preferably less than 1% by weight chloride ions, or chloride containing compounds.

The detergent composition can comprise a coated source of percarbonate. The source of percarbonate can be coated by any coating known in the art, or combination thereof. Preferably, the coating comprises one or more alkali metal salt, preferably this is sodium carbonate, sodium bicarbonate, sodium silicate, sodium sulphate, or any combination thereof. The coated sodium percarbonate is formed by contacting the percarbonate source with the coating in such a way that the coating is present on the surface of the source of percarbonate. The coating is present on the surface of the source of percarbonate in such a way as to either completely or partially coat the percarbonate source.

The coated source of percarbonate is prepared by a method comprising contacting the source of percarbonate with carbon dioxide to form a coating of sodium bicarbonate on the surface of the sodium percarbonate particle. Usually this is done in the presence of moisture.

The coated source of percarbonate particle comprises a coating present at a level of 0.01% to 20%, preferably 1% to 15%, more preferably 4% to 10%, by weight of the coated sodium percarbonate particle Any detergent components which comprise sodium percarbonate can also be coated in this manner.

The coated percarbonate can be prepared by a fluidised bed process and/or by spraying the coating onto the surface of the sodium percarbonate.

The source of percarbonate preferably has a mean particle size of 500 to 1000, preferably 500 to 850, more preferably 550 to 800 micrometers.

Additional detergent ingredients

The detergent composition typically comprises additional detergent ingredients. These detergent ingredients are described below. Preferably the detergent composition comprises a surfactant, builder, chelant and a bleaching system.

Surfactants

Nonionic Alkoxylated Surfactant

Essentially any alkoxylated nonionic surfactants can also be comprised by the composition of the invention. The ethoxylated and propoxylated nonionic surfactants are preferred. Preferred alkoxylated surfactants can be selected from the classes of the nonionic condensates of alkyl phenols, nonionic ethoxylated alcohols, nonionic ethoxylated/propoxylated fatty alcohols, nonionic ethoxylate/propoxylate condensates with propylene glycol, and the nonionic ethoxylate condensation products with propylene oxide/ethylene diamine adducts.

Highly preferred are nonionic alkoxylated alcohol surfactants, being the condensation products of aliphatic alcohols with from 1 to 75 moles of alkylene oxide, in particular about 50 or from 1 to 15 moles, preferably to 11 moles, particularly ethylene oxide and/or propylene oxide, are highly preferred nonionic surfactant comprised in the anhydrous component of the particles of the invention. The alkyl chain of the aliphatic alcohol can either be straight or branched, primary or secondary, and generally contains from 6 to 22 carbon atoms. Particularly preferred are the condensation products of alcohols having an alkyl group containing from 8 to 20 carbon atoms with from 2 to 9 moles and in particular 3 or 5 moles, of ethylene oxide per mole of alcohol.

Nonionic Polyhydroxy Fatty Acid Amide Surfactant

Polyhydroxy fatty acid amides are highly preferred nonionic surfactant comprised by the composition of the invention , in particular those having the structural formula R^2CONR^1Z wherein : R1 is H, C_{1-18}, preferably C_1-C_4 hydrocarbyl, 2-hydroxy ethyl, 2-hydroxy propyl, ethoxy, propoxy, or a mixture thereof, preferable C1-C4 alkyl, more preferably C_1 or C_2 alkyl, most preferably C_1 alkyl (i.e., methyl); and R_2 is a C_5-C_{31} hydrocarbyl, preferably straight-chain C_5-C_{19} or C_7-C_{19} alkyl or alkenyl, more preferably straight-chain C_9-C_{17} alkyl or alkenyl, most preferably straight-chain C_{11}-

C_{17} alkyl or alkenyl, or mixture thereof; and Z is a polyhydroxyhydrocarbyl having a linear hydrocarbyl chain with at least 3 hydroxyls directly connected to the chain, or an alkoxylated derivative (preferably ethoxylated or propoxylated) thereof. Z preferably will be derived from a reducing sugar in a reductive amination reaction; more preferably Z is a glycityl.

A highly preferred nonionic polyhydroxy fatty acid amide surfactant for use herein is a C_{12}-C_{14} , a C_{15}-C_{17} and/or C_{16}-C_{18} alkyl N-methyl glucamide.

It may be particularly preferred that the composition of the invention comprises a mixture of a C_{12}-C_{18} alkyl N-methyl glucamide and condensation products of an alcohol having an alkyl group containing from 8 to 20 carbon atoms with from 2 to 9 moles and in particular 3 or 5 moles, of ethylene oxide per mole of alcohol.

The polyhydroxy fatty acid amide can be prepared by any suitable process. One particularly preferred process is described in detail in WO 9206984. A product comprising about 95% by weight polyhydroxy fatty acid amide, low levels of undesired impurities such as fatty acid esters and cyclic amides, and which is molten typically above about 80°C, can be made by this process.

Nonionic Fatty Acid Amide Surfactant

Fatty acid amide surfactants or alkoxylated fatty acid amides can also be comprised by the composition of the invention. They include those having the formula: $R^6CON(R^7)(R^8)$ wherein R^6 is an alkyl group containing from 7 to 21, preferably from 9 to 17 carbon or even 11 to 13 carbon atoms and R^7 and R^8 are each individually selected from the group consisting of hydrogen, C_1-C_4 alkyl, C_1-C_4 hydroxyalkyl, and $-(C_2H_4O)_xH$, where x is in the range of from 1 to 11, preferably 1 to 7, more preferably form 1-5, whereby it may be preferred that R^7 is different to R^8, one having x being 1 or 2, one having x being from 3 to 11 or preferably 5.

Nonionic Alkyl Esters of Fatty Acid Surfactant

Alkyl esters of fatty acids can also be comprised in any material of the invention. They include those having the formula: $R^9COO(R^{10})$ wherein R^9 is an alkyl group containing from 7 to 21, preferably from 9 to 17 carbon or even 11 to 13 carbon atoms and R^{10} is a C_1-C_4 alkyl, C_1-C_4 hydroxyalkyl, or $-(C_2H_4O)_xH$, where x is in the range of from 1 to 11, preferably 1 to 7, more preferably form 1-5, whereby it may be preferred that R^{10} is a methyl or ethyl group.

Nonionic Alkylpolysaccharide Surfactant

Alkylpolysaccharides can also be comprised by the composition of the invention, such as those disclosed in US Patent 4,565,647, Llenado, issued January 21, 1986, having a hydrophobic group containing from 6 to 30 carbon atoms and a polysaccharide, e.g., a polyglycoside, hydrophilic group containing from 1.3 to 10 saccharide units.

Preferred alkylpolyglycosides have the formula

$$R^2O(C_nH_{2n}O)t(\text{glycosyl})_x$$

wherein R^2 is selected from the group consisting of alkyl, alkylphenyl, hydroxyalkyl, hydroxyalkylphenyl, and mixtures thereof in which the alkyl groups contain from 10 to 18 carbon atoms; n is 2 or 3; t is from 0 to 10, and x is from 1.3 to 8. The glycosyl is preferably derived from glucose.

Polyethylene/ Propylene Glycols

The composition of the invention may comprise polyethylene and/or propylene glycol, particularly those of molecular weight 1000-10000, more particularly 2000 to 8000 and most preferably about 4000.

Anionic Surfactant

The detergent composition of the invention, preferably comprises one or more anionic surfactants. Any anionic surfactant useful for detersive purposes is suitable. Examples include salts (including, for example, sodium, potassium, ammonium, and substituted ammonium salts such as mono-, di- and triethanolamine salts) of the anionic sulphate, sulphonate, carboxylate and sarcosinate surfactants. Anionic sulphate surfactants are preferred.

Other anionic surfactants include the isethionates such as the acyl isethionates, N-acyl taurates, fatty acid amides of methyl tauride, alkyl succinates and sulfosuccinates, monoesters of sulfosuccinate (especially saturated and unsaturated C_{12}-C_{18} monoesters) diesters of sulfosuccinate (especially saturated and unsaturated C_6-C_{14} diesters), N-acyl sarcosinates. Resin acids and hydrogenated resin acids are also suitable, such as rosin, hydrogenated rosin, and resin acids and hydrogenated resin acids present in or derived from tallow oil.

Anionic Sulphate Surfactant

Anionic sulphate surfactants suitable for use herein include the linear and branched primary and secondary alkyl sulphates, alkyl ethoxysulphates, fatty oleoyl glycerol sulphates, alkyl phenol ethylene oxide ether sulphates, the C_5-C_{17} acyl-N-(C_1-C_4 alkyl) and -N-(C_1-C_2 hydroxyalkyl) glucamine sulphates, and sulphates of alkylpolysaccharides such as the sulphates of alkylpolyglucoside (the nonionic non-sulphated compounds being described herein).

Alkyl sulphate surfactants are preferably selected from the linear and branched primary C_9-C_{22} alkyl sulphates, more preferably the C_{11}-C_{15} branched chain alkyl sulphates and the C_{12}-C_{14} linear chain alkyl sulphates.

Alkyl ethoxysulfate surfactants are preferably selected from the group consisting of the C_{10}-C_{18} alkyl sulphates which have been ethoxylated with from 0.5 to 50 moles of ethylene oxide per molecule. More preferably, the alkyl ethoxysulfate surfactant is a C_{11}-C_{18}, most preferably C_{11}-C_{15} alkyl sulphate which has been ethoxylated with from 0.5 to 7, preferably from 1 to 5, moles of ethylene oxide per molecule.

Anionic Sulphonate Surfactant

Anionic sulphonate surfactants suitable for use herein include the salts of C_5-C_{20} linear or branched alkylbenzene sulphonates, alkyl ester sulphonates, in particular methyl ester sulphonates, C_6-C_{22} primary or secondary alkane sulphonates, C_6-C_{24} olefin sulphonates, sulphonated polycarboxylic acids, alkyl glycerol sulphonates, fatty acyl glycerol sulphonates, fatty oleyl glycerol sulphonates, and any mixtures thereof.

Anionic Carboxylate Surfactant

Suitable anionic carboxylate surfactants include the alkyl ethoxy carboxylates, the alkyl polyethoxy polycarboxylate surfactants and the soaps ('alkyl carboxyls'), especially certain secondary soaps as described herein.

Suitable alkyl ethoxy carboxylates include those with the formula $RO(CH_2CH_20)_x CH_2C00^-M^+$ wherein R is a C_6 to C_{18} alkyl group, x ranges from O to 10, and the ethoxylate distribution is such that, on a weight basis, the amount of material where x is 0 is less than 20 % and M is a cation. Suitable alkyl polyethoxy polycarboxylate surfactants include those having the formula $RO\text{-}(CHR_1\text{-}CHR_2\text{-}O)_X\text{-}R_3$ wherein R is a C_6 to C_{18} alkyl group, x is from 1 to 25, R_1 and R_2 are selected from the group consisting of hydrogen, methyl acid radical, succinic acid radical, hydroxysuccinic acid radical, and mixtures thereof, and R_3 is selected from the group consisting of hydrogen, substituted or unsubstituted hydrocarbon having between 1 and 8 carbon atoms, and mixtures thereof.

Suitable soap surfactants include the secondary soap surfactants which contain a carboxyl unit connected to a secondary carbon. Preferred secondary soap surfactants for use herein are water-soluble members selected from the group consisting of the water-soluble salts of 2-methyl-1-undecanoic acid, 2-ethyl-1-decanoic acid, 2-propyl-1-nonanoic acid, 2-butyl-1-octanoic acid and 2-pentyl-1-heptanoic acid. Certain soaps may also be included as suds suppressers.

Alkali Metal Sarcosinate Surfactant

Other suitable anionic surfactants are the alkali metal sarcosinates of formula $R\text{-}CON(R^1)CH_2COOM$, wherein R is a $C_5\text{-}C_{17}$ linear or branched alkyl or alkenyl group, R^1 is a $C_1\text{-}C_4$ alkyl group and M is an alkali metal ion. Preferred examples are the myristyl and oleoyl methyl sarcosinates in the form of their sodium salts.

Cationic Surfactant

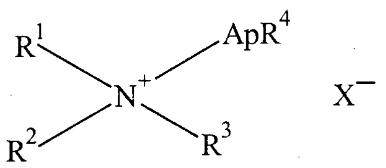
Another preferred optional ingredient of the detergent composition of the invention, is a cationic surfactant, which may preferably be present at a level of from 0.1% to 60% by weight of the composition or particle, more preferably from 0.4% to 20%, most preferably from 0.5% to 5% by weight of the composition.

When present, the ratio of the anionic surfactant to the cationic surfactant is preferably from 25:1 to 1:3, more preferably from 15:1 to 1:1. most preferably from 10:1 to 1:1 The ratio of cationic surfactant to the stabilising agent is preferably from 1:30 to 20:1, more preferably from 1:20 to 10:1.

Preferably the cationic surfactant is selected from the group consisting of cationic ester surfactants, cationic mono-alkoxylated amine surfactants, cationic bis-alkoxylated amine surfactants and mixtures thereof.

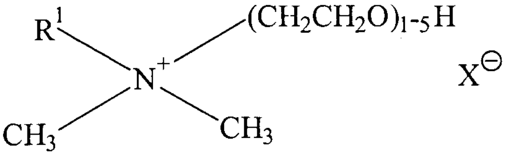
Cationic Mono-Alkoxylated Amine Surfactants

The optional cationic mono-alkoxylated amine surfactant for use herein, has the general formula:



wherein R^1 is an alkyl or alkenyl moiety containing from about 6 to about 18 carbon atoms, preferably 6 to about 16 carbon atoms, most preferably from about 6 to about 11 carbon atoms; R^2 and R^3 are each independently alkyl groups containing from one to about three carbon atoms, preferably methyl; R^4 is selected from hydrogen (preferred), methyl and ethyl, X^- is an anion such as chloride, bromide, methylsulphate, sulphate, or the like, to provide electrical neutrality; A is selected from C_1-C_4 alkoxy, especially ethoxy (i.e., $-CH_2CH_2O-$), propoxy, butoxy and mixtures thereof; and p is from 1 to about 30, preferably 1 to about 15, most preferably 1 to about 8.

Highly preferred cationic mono-alkoxylated amine surfactants for use herein are of the formula:

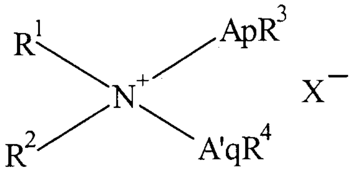


wherein R^1 is C_6-C_{18} hydrocarbyl and mixtures thereof, preferably C_6-C_{14}, especially C_6-C_{11} alkyl, preferably C_8 and C_{10} alkyl, and X is any convenient anion to provide charge balance, preferably chloride or bromide.

As noted, compounds of the foregoing type include those wherein the ethoxy (CH_2CH_2O) units (EO) are replaced by butoxy, isopropoxy [$CH(CH_3)CH_2O$] and [$CH_2CH(CH_3O$] units (i-Pr) or n-propoxy units (Pr), or mixtures of EO and/or Pr and/or i-Pr units.

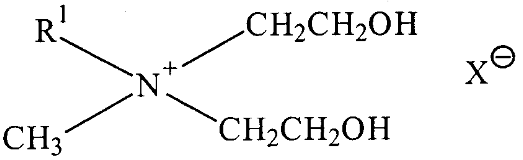
Cationic Bis-Alkoxylated Amine Surfactant

The cationic bis-alkoxylated amine surfactant for use herein, has the general formula:



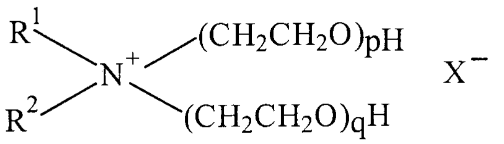
wherein R^1 is an alkyl or alkenyl moiety containing from about 6 to about 18 carbon atoms, preferably 6 to about 16 carbon atoms, more preferably 6 to about 11, most preferably from about 8 to about 10 carbon atoms; R^2 is an alkyl group containing from one to three carbon atoms, preferably methyl; R^3 and R^4 can vary independently and are selected from hydrogen (preferred), methyl and ethyl, X^- is an anion such as chloride, bromide, methylsulphate, sulphate, or the like, sufficient to provide electrical neutrality. A and A' can vary independently and are each selected from C_1-C_4 alkoxy, especially ethoxy, (i.e., $-CH_2CH_2O-$), propoxy, butoxy and mixtures thereof; p is from 1 to about 30, preferably 1 to about 4 and q is from 1 to about 30, preferably 1 to about 4, and most preferably both p and q are 1.

Highly preferred cationic bis-alkoxylated amine surfactants for use herein are of the formula:



wherein R^1 is C_6-C_{18} hydrocarbyl and mixtures thereof, preferably C_6, C_8, C_{10}, C_{12}, C_{14} alkyl and mixtures thereof. X is any convenient anion to provide charge balance, preferably chloride. With reference to the general cationic bis-alkoxylated amine structure noted above, since in a preferred compound R^1 is derived from (coconut) C_{12}-C_{14} alkyl fraction fatty acids, R^2 is methyl and ApR^3 and $A'qR^4$ are each monoethoxy.

Other cationic bis-alkoxylated amine surfactants useful herein include compounds of the formula:



wherein R^1 is C_6-C_{18} hydrocarbyl, preferably C_6-C_{14} alkyl, independently p is 1 to about 3 and q is 1 to about 3, R^2 is C_1-C_3 alkyl, preferably methyl, and X is an anion, especially chloride or bromide.

Other compounds of the foregoing type include those wherein the ethoxy (CH_2CH_2O) units (EO) are replaced by butoxy (Bu) isopropoxy [$CH(CH_3)CH_2O$] and [$CH_2CH(CH_3O$] units (i-Pr) or n-propoxy units (Pr), or mixtures of EO and/or Pr and/or i-Pr units.

Amphoteric Surfactant

Suitable amphoteric surfactants for use herein include the amine oxide surfactants and the alkyl amphocarboxylic acids.

Suitable amine oxides include those compounds having the formula $R^3(OR^4)_xN^0(R^5)_2$ wherein R^3 is selected from an alkyl, hydroxyalkyl, acylamidopropoyl and alkyl phenyl group, or mixtures thereof, containing from 8 to 26 carbon atoms; R^4 is an alkylene or hydroxyalkylene group containing from 2 to 3 carbon atoms, or mixtures thereof; x is from 0 to 5, preferably from 0 to 3; and each R^5 is an alkyl or hydroxyalkyl group containing from 1 to 3, or a polyethylene oxide group containing from 1 to 3 ethylene oxide groups. Preferred are C_{10}-C_{18} alkyl dimethylamine oxide, and C_{10-18} acylamido alkyl dimethylamine oxide.

A suitable example of an alkyl aphodicarboxylic acid is Miranol(TM) C2M Conc. manufactured by Miranol, Inc., Dayton, NJ.

Zwitterionic Surfactant

Zwitterionic surfactants can also be comprised by the composition of the invention. These surfactants can be broadly described as derivatives of secondary and tertiary amines, derivatives of heterocyclic secondary and tertiary amines, or derivatives of quaternary ammonium, quaternary phosphonium or tertiary sulfonium compounds. Betaine and sultaine surfactants are exemplary zwitterionic surfactants for use herein.

Suitable betaines are those compounds having the formula $R(R')_2N^+R^2COO^-$ wherein R is a C_6-C_{18} hydrocarbyl group, each R^1 is typically C_1-C_3 alkyl, and R^2 is a C_1-C_5 hydrocarbyl group. Preferred betaines are C_{12-18} dimethyl-ammonio hexanoate and the C_{10-18} acylamidopropane (or ethane) dimethyl (or diethyl) betaines. Complex betaine surfactants are also suitable for use herein.

Water-Soluble Builder Compound

The detergent composition of the invention preferably comprises a water-soluble builder compound, typically present at a level of from 0% to 36% by weight, preferably from 1% to 35% by weight, more preferably from 10% to 35%, even more preferably from 12% to 30% by weight of the composition or particle. Preferably, the water-soluble builder compound is an alkali or earth alkali metal salt of phosphate present at the level described above.

Other typical water-soluble builder compounds include the water soluble monomeric polycarboxylates, or their acid forms, homo or copolymeric polycarboxylic acids or their salts in which the polycarboxylic acid comprises at least two carboxylic radicals separated from each other by not more that two carbon atoms, borates, phosphates, and mixtures of any of the foregoing.

The carboxylate or polycarboxylate builder can be monomeric or oligomeric in type although monomeric polycarboxylates are generally preferred for reasons of cost and performance.

Suitable carboxylates containing one carboxy group include the water soluble salts of lactic acid, glycolic acid and ether derivatives thereof. Polycarboxylates containing two carboxy groups include the water-soluble salts of succinic acid, malonic acid, (ethylenedioxy) diacetic acid, maleic acid, diglycolic acid, tartaric acid, tartronic acid and fumaric acid, as well as the ether carboxylates and the sulfinyl carboxylates. Polycarboxylates containing three carboxy groups include, in particular, water-soluble citrates, aconitrates and citraconates as well as succinate derivatives such as the carboxymethyloxysuccinates described in British Patent No. 1,379,241, lactoxysuccinates described in British Patent No. 1,389,732, and aminosuccinates described in Netherlands Application 7205873, and the oxypolycarboxylate materials such as 2-oxa-1,1,3-propane tricarboxylates described in British Patent No. 1,387,447.

Polycarboxylates containing four carboxy groups include oxydisuccinates disclosed in British Patent No. 1,261,829, 1,1,2,2-ethane tetracarboxylates, 1,1,3,3-propane tetracarboxylates and 1,1,2,3-propane tetracarboxylates. Polycarboxylates containing sulfo substituents include the sulfosuccinate derivatives disclosed in British Patent Nos. 1,398,421 and 1,398,422 and in US Patent No. 3,936,448, and the sulphonated pyrolysed citrates described in British Patent No. 1,439,000. Preferred polycarboxylates are hydroxycarboxylates containing up to three carboxy groups per molecule, more particularly citrates.

Borate builders, as well as builders containing borate-forming materials that can produce borate under detergent storage or wash conditions are useful water-soluble builders herein.

Suitable examples of water-soluble phosphate builders are the alkali metal tripolyphosphates, sodium, potassium and ammonium pyrophosphate, sodium and potassium and ammonium pyrophosphate, sodium and potassium orthophosphate, sodium polymeta/phosphate in which the degree of polymerization ranges from about 6 to 21, and salts of phytic acid.

Partially Soluble or Insoluble Builder Compound

The detergent composition of the invention, or any detergent component comprised by the detergent composition of the invention, may contain a partially soluble or insoluble builder compound.

Examples of largely water insoluble builders include the sodium aluminosilicates.

Suitable aluminosilicate zeolites have the unit cell formula $Na_z[(AlO_2)_z(SiO_2)y]. xH_2O$ wherein z and y are at least 6; the molar ratio of z to y is from 1.0 to 0.5 and x is at least 5, preferably from 7.5 to 276, more preferably from 10 to 264. The aluminosilicate material are in hydrated form and are preferably crystalline, containing from 10% to 28%, more preferably from 18% to 22% water in bound form.

The aluminosilicate zeolites can be naturally occurring materials, but are preferably synthetically derived. Synthetic crystalline aluminosilicate ion exchange materials are available under the designations Zeolite A, Zeolite B, Zeolite P, Zeolite X, Zeolite HS and mixtures thereof. Zeolite A has the formula:

$Na_{12} [AlO_2)_{12} (SiO_2)_{12}]. xH_2O$

wherein x is from 20 to 30, especially 27. Zeolite X has the formula $Na_{86} [(AlO_2)_{86}(SiO_2)_{106}]. 276\ H_2O$.

Preferred crystalline layered silicates for use herein have the general formula:

$$NaMSi_xO_{2x+1}.yH_2O$$

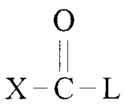
wherein M is sodium or hydrogen, x is a number from 1.9 to 4 and y is a number from 0 to 20. Crystalline layered sodium silicates of this type are disclosed in EP-A-0164514 and methods for their preparation are disclosed in DE-A-3417649 and DE-A-3742043. Herein, x in the general formula above preferably has a value of 2, 3 or 4 and is preferably 2. The most preferred material is $\delta\text{-}Na_2Si_2O_5$, available from Hoechst AG as NaSKS-6.

Organic Peroxyacid Bleaching System

The detergent composition of the invention preferably comprises an organic peroxyacid precursor. The production of the organic peroxyacid may occur by an in situ reaction of such a precursor with the percarbonate source. In an alternative preferred execution a preformed organic peroxyacid is incorporated directly into the composition.

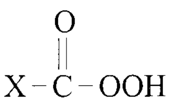
Peroxyacid Bleach Precursor

Peroxyacid bleach precursors are compounds which react with hydrogen peroxide in a perhydrolysis reaction to produce a peroxyacid. Generally peroxyacid bleach precursors may be represented as:



$$X-\overset{\overset{\large O}{\|}}{C}-L$$

where L is a leaving group and X is essentially any functionality, such that on perhydroloysis the structure of the peroxyacid produced is:



$$X-\overset{\overset{\large O}{\|}}{C}-OOH$$

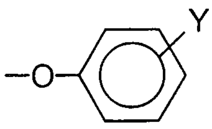
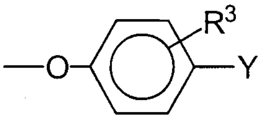
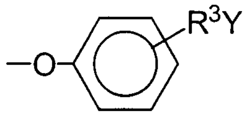
Suitable peroxyacid bleach precursor compounds typically contain one or more N- or O-acyl groups, which precursors can be selected from a wide range of classes. Suitable classes include anhydrides, esters, imides, lactams and acylated derivatives of imidazoles and oximes. Examples of useful materials within these classes are disclosed in GB-A-1586789. Suitable esters are disclosed in GB-A-836988, 864798, 1147871, 2143231 and

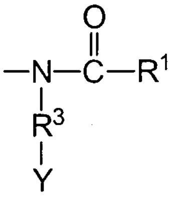
EP-A-0170386.

Leaving Groups

The leaving group, hereinafter L group, must be sufficiently reactive for the perhydrolysis reaction to occur within the optimum time frame (e.g., a wash cycle). However, if L is too reactive, this activator will be difficult to stabilize for use in a bleaching composition.

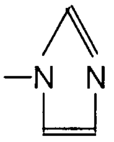
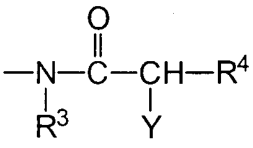
Preferred L groups are selected from the group consisting of:


,

, and




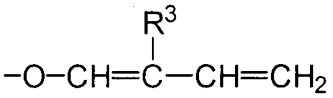
$$-\underset{\underset{\underset{Y}{|}}{R^3}}{\underset{|}{N}}-\overset{\overset{O}{\|}}{C}-R^1$$

,


,


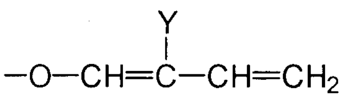
$$-\underset{R^3}{\underset{|}{N}}-\overset{\overset{O}{\|}}{C}-\underset{Y}{\underset{|}{CH}}-R^4$$

,



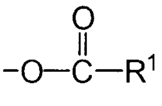
$$-O-CH{=}\overset{\overset{R^3}{|}}{C}-CH{=}CH_2$$

,



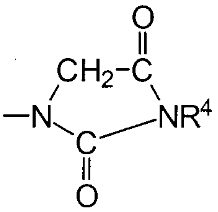
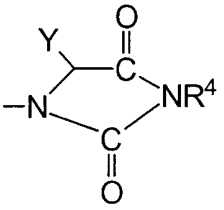
$$-O-CH{=}\overset{\overset{Y}{|}}{C}-CH{=}CH_2$$

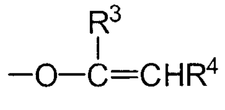
,



$$-O-\overset{\overset{O}{\|}}{C}-R^1$$

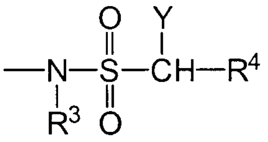
,


,

,



$-O-\overset{R^3}{\overset{|}{C}}=CHR^4$

, and



$-\underset{R^3}{\underset{|}{N}}-\overset{O}{\overset{\|}{\underset{O}{\underset{\|}{S}}}}-\overset{Y}{\overset{|}{CH}}-R^4$

and mixtures thereof, wherein R^1 is an alkyl, aryl, or alkaryl group containing from 1 to 14 carbon atoms, R^3 is an alkyl chain containing from 1 to 8 carbon atoms, R^4 is H or R^3, and Y is H or a solubilizing group. Any of R^1, R^3 and R^4 may be substituted by essentially any functional group including, for example alkyl, hydroxy, alkoxy, halogen, amine, nitrosyl, amide and ammonium or alkyl ammonium groups.

The preferred solubilizing groups are $-SO_3^-M^+$, $-CO_2^-M^+$, $-SO_4^-M^+$, $-N^+(R^3)_4X^-$ and O<--$N(R^3)_3$ and most preferably $-SO_3^-M^+$ and $-CO_2^-M^+$ wherein R^3 is an alkyl chain containing from 1 to 4 carbon atoms, M is a cation which provides solubility to the bleach activator and X is an anion which provides solubility to the bleach activator. Preferably, M is an alkali metal, ammonium or substituted ammonium cation, with sodium and potassium being most preferred, and X is a halide, hydroxide, methylsulphate or acetate anion.

Alkyl Percarboxylic Acid Bleach Precursors

Alkyl percarboxylic acid bleach precursors form percarboxylic acids on perhydrolysis. Preferred precursors of this type provide peracetic acid on perhydrolysis.

Typical alkyl percarboxylic precursor compounds of the imide type include the N-,N,N^1N^1 tetra acetylated alkylene diamines wherein the alkylene group contains from 1 to 6 carbon atoms, particularly those compounds in which the alkylene group contains 1, 2 and 6 carbon atoms. Tetraacetyl ethylene diamine (TAED) is particularly preferred. The TAED is preferably not present in the agglomerated particle of the present invention, but preferably present in the detergent composition, comprising the particle.

Other alkyl percarboxylic acid precursors include sodium 3,5,5-tri-methyl hexanoyloxybenzene sulphonate (iso-NOBS), sodium nonanoyloxybenzene sulphonate (NOBS), sodium acetoxybenzene sulphonate (ABS) and pentaacetyl glucose.

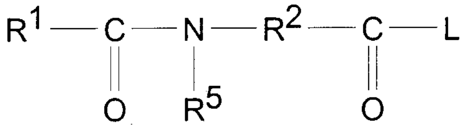
The detergent composition preferably comprises 0.1% to 2%, preferably 0.1% to 1%, more preferably 0.2% to 0.7%, even more preferably 0.25% to 0.6%, by weight TAED.

The detergent composition preferably comprises 0.1% to 4%, preferably 0.2% to 3%, more preferably 0.4% to 2%, even more preferably 0.7% to 1.9%, by weight NOBS.

The detergent composition can also comprise a combination of NOBS and TAED, present at the levels described hereinbefore.

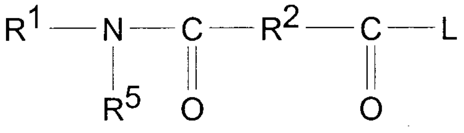
<u>Amide Substituted Alkyl Peroxyacid Precursors</u>

Amide substituted alkyl peroxyacid precursor compounds are suitable herein, including those of the following general formulae:



$$R^1-\underset{\underset{O}{\|}}{C}-\underset{\underset{R^5}{|}}{N}-R^2-\underset{\underset{O}{\|}}{C}-L$$

or



$$R^1-\underset{\underset{R^5}{|}}{N}-\underset{\underset{O}{\|}}{C}-R^2-\underset{\underset{O}{\|}}{C}-L$$

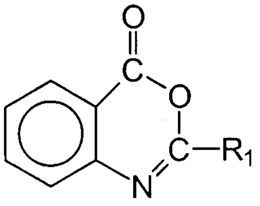
wherein R^1 is an alkyl group with from 1 to 14 carbon atoms, R^2 is an alkylene group containing from 1 to 14 carbon atoms, and R^5 is H or an alkyl group containing 1 to 10 carbon atoms and L can be essentially any leaving group. Amide substituted bleach activator compounds of this type are described in EP-A-0170386.

<u>Perbenzoic Acid Precursor</u>

Perbenzoic acid precursor compounds provide perbenzoic acid on perhydrolysis. Suitable O-acylated perbenzoic acid precursor compounds include the substituted and unsubstituted benzoyl oxybenzene sulphonates, and the benzoylation products of sorbitol, glucose, and all saccharides with benzoylating agents, and those of the imide type including N-benzoyl succinimide, tetrabenzoyl ethylene diamine and the N-benzoyl substituted ureas. Suitable imidazole type perbenzoic acid precursors include N-benzoyl imidazole and N-benzoyl benzimidazole. Other useful N-acyl group-containing perbenzoic acid precursors include N-benzoyl pyrrolidone, dibenzoyl taurine and benzoyl pyroglutamic acid.

Benzoxazin Organic Peroxyacid Precursors

Also suitable are precursor compounds of the benzoxazin-type, as disclosed for example in EP-A-332,294 and EP-A-482,807, particularly those having the formula:

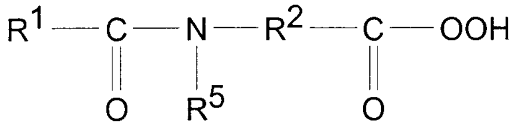


wherein R_1 is H, alkyl, alkaryl, aryl, or arylalkyl.

Preformed Organic Peroxyacid

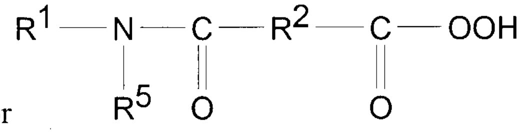
The organic peroxyacid bleaching system may contain a preformed organic peroxyacid.

A preferred class of organic peroxyacid compounds are the amide substituted compounds of the following general formulae:



$$R^1-\underset{\displaystyle O}{\underset{\|}{C}}-\underset{\displaystyle R^5}{\underset{|}{N}}-R^2-\underset{\displaystyle O}{\underset{\|}{C}}-OOH$$

or



$$R^1-\underset{\displaystyle R^5}{\underset{|}{N}}-\underset{\displaystyle O}{\underset{\|}{C}}-R^2-\underset{\displaystyle O}{\underset{\|}{C}}-OOH$$

wherein R^1 is an alkyl, aryl or alkaryl group with from 1 to 14 carbon atoms, R^2 is an alkylene, arylene, and alkarylene group containing from 1 to 14 carbon atoms, and R^5 is H or an alkyl, aryl, or alkaryl group containing 1 to 10 carbon atoms. Amide substituted organic peroxyacid compounds of this type are described in EP-A-0170386.

Other organic peroxyacids include diacyl and tetraacylperoxides, especially diperoxydodecanedioc acid, diperoxytetradecanedioc acid and diperoxyhexadecanedioc acid. Mono- and diperazelaic acid, mono- and diperbrassylic acid and N-phthaloylaminoperoxicaproic acid are also suitable herein.

Heavy Metal Ion Sequestrant

The detergent composition of the invention, or any detergent component comprised by the detergent composition of the invention, preferably comprise as an optional detergent ingredient, a heavy metal ion sequestrant. By heavy metal ion sequestrant it is meant herein components which act to sequester (chelate) heavy metal ions. These components may also have calcium and magnesium chelation capacity, but preferentially they show selectivity to binding heavy metal ions such as iron, manganese and copper.

Heavy metal ion sequestrants are generally present at a level of from 0.05% to 2%, preferably from 0.1% to 1.5%, more preferably from 0.25% to 1.2% and most preferably from 0.5% to 1% by weight of the composition.

Suitable heavy metal ion sequestrants for use herein include organic phosphonates, such as the amino alkylene poly (alkylene phosphonates), alkali metal ethane 1-hydroxy disphosphonates and nitrilo trimethylene phosphonates.

Preferred among the above species are diethylene triamine penta (methylene phosphonate), ethylene diamine tri (methylene phosphonate) hexamethylene diamine tetra (methylene phosphonate) and hydroxy-ethylene 1,1 diphosphonate.

Other suitable heavy metal ion sequestrant for use herein include nitrilotriacetic acid and polyaminocarboxylic acids such as ethylenediaminotetracetic acid, ethylenetriamine pentacetic acid, ethylenediamine disuccinic acid, ethylenediamine diglutaric acid, 2-hydroxypropylenediamine disuccinic acid or any salts thereof. Especially preferred is ethylenediamine-N,N'-disuccinic acid (EDDS) or the alkali metal, alkaline earth metal, ammonium, or substituted ammonium salts thereof, or mixtures thereof.

Other suitable heavy metal ion sequestrants for use herein are iminodiacetic acid derivatives such as 2-hydroxyethyl diacetic acid or glyceryl imino diacetic acid, described in EP-A-317,542 and EP-A-399,133. The iminodiacetic acid-N-2-hydroxypropyl sulfonic acid and aspartic acid N-carboxymethyl N-2-hydroxypropyl-3-sulfonic acid sequestrants described in EP-A-516,102 are also suitable herein. The β-alanine-N,N'-diacetic acid, aspartic acid-N,N'-diacetic acid, aspartic acid-N-monoacetic acid and iminodisuccinic acid sequestrants described in EP-A-509,382 are also suitable.

EP-A-476,257 describes suitable amino based sequestrants. EP-A-510,331 describes suitable sequestrants derived from collagen, keratin or casein. EP-A-528,859 describes a suitable alkyl iminodiacetic acid sequestrant. Dipicolinic acid and 2-phosphonobutane-1,2,4-tricarboxylic acid are also suitable. Glycinamide-N,N'-disuccinic acid (GADS), ethylenediamine-N-N'-diglutaric acid (EDDG) and 2-hydroxypropylenediamine-N-N'-disuccinic acid (HPDDS) are also suitable.

Enzyme

Another preferred optional detergent ingredient useful in the detergent composition of the invention, or any detergent component comprised by the detergent composition of the invention, is one or more additional enzymes.

Preferred additional enzymatic materials include the commercially available lipases, cutinases, amylases, neutral and alkaline proteases, esterases, cellulases, pectinases, lactases and peroxidases conventionally incorporated into detergent compositions. Suitable enzymes are discussed in US Patents 3,519,570 and 3,533,139.

Preferred commercially available protease enzymes include those sold under the tradenames Alcalase, Savinase, Primase, Durazym, and Esperase by Novo Industries A/S (Denmark), those sold under the tradename Maxatase, Maxacal and Maxapem by Gist-Brocades, those sold by Genencor International, and those sold under the tradename Opticlean and Optimase by Solvay Enzymes. Protease enzyme may be incorporated into the compositions in accordance with the invention at a level of from 0.0001% to 4% active enzyme by weight of the composition.

Preferred amylases include, for example, α-amylases obtained from a special strain of B licheniformis, described in more detail in GB-1,269,839 (Novo). Preferred commercially available amylases include for example, those sold under the tradename Rapidase by Gist-Brocades, and those sold under the tradename Termamyl and BAN by Novo Industries A/S. Amylase enzyme may be incorporated into the composition in accordance with the invention at a level of from 0.0001% to 2% active enzyme by weight of the composition.

Lipolytic enzyme may be present at levels of active lipolytic enzyme of from 0.0001% to 10% by weight of the particle, preferably 0.001% to 3% by weight of the composition, most preferably from 0.001% to 0.5% by weight of the compositions.

The lipase may be fungal or bacterial in origin being obtained, for example, from a lipase producing strain of Humicola sp., Thermomyces sp. or Pseudomonas sp. including Pseudomonas pseudoalcaligenes or Pseudomas fluorescens. Lipase from chemically or genetically modified mutants of these strains are also useful herein. A preferred lipase is derived from Pseudomonas pseudoalcaligenes, which is described in Granted European

Patent, EP-B-0218272.

Another preferred lipase herein is obtained by cloning the gene from Humicola lanuginosa and expressing the gene in Aspergillus oryza, as host, as described in European Patent Application, EP-A-0258 068, which is commercially available from Novo Industri A/S, Bagsvaerd, Denmark, under the trade name Lipolase. This lipase is also described in U.S. Patent 4,810,414, Huge-Jensen et al, issued March 7, 1989.

Organic Polymeric Compound

Organic polymeric compounds are preferred additional detergent ingredients of the detergent composition of the invention, or any detergent component comprised by the detergent composition of the invention, and are preferably present as components of any particulate components where they may act such as to bind the particulate component together. By organic polymeric compound it is meant herein essentially any polymeric organic compound commonly used as dispersants, and anti-redeposition and soil suspension agents in detergent compositions, including any of the high molecular weight organic polymeric compounds described as clay flocculating agents herein.

Organic polymeric compound is typically incorporated in the detergent compositions of the invention at a level of from 0.1% to 50% by weight of the particle, preferably from 0.5% to 25%, most preferably from 1% to 15% by weight of the compositions.

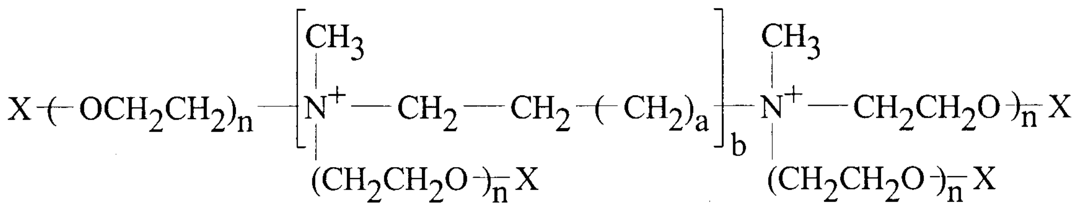
Examples of organic polymeric compounds include the water soluble organic homo- or co-polymeric polycarboxylic acids or their salts in which the polycarboxylic acid comprises at least two carboxyl radicals separated from each other by not more than two carbon atoms. Polymers of the latter type are disclosed in GB-A-1,596,756. Examples of such salts are polyacrylates of MWt 2000-5000 and their copolymers with maleic anhydride, such copolymers having a molecular weight of from 20,000 to 100,000, especially 40,000 to 80,000.

The polyamino compounds are useful herein including those derived from aspartic acid such as those disclosed in EP-A-305282, EP-A-305283 and EP-A-351629.

Terpolymers containing monomer units selected from maleic acid, acrylic acid, polyaspartic acid and vinyl alcohol, particularly those having an average molecular weight of from 5,000 to 10,000, are also suitable herein.

Other organic polymeric compounds suitable for incorporation in the detergent compositions herein include cellulose derivatives such as methylcellulose, carboxymethylcellulose, hydroxypropylmethylcellulose and hydroxyethylcellulose.

Another organic compound, which is a preferred clay dispersant/ anti-redeposition agent, for use herein, can be the ethoxylated cationic monoamines and diamines of the formula:



$$X(OCH_2CH_2)_n-\left[\underset{(CH_2CH_2O)_nX}{\overset{CH_3}{N^+}}-CH_2-CH_2(CH_2)_a\right]_b-\underset{(CH_2CH_2O)_nX}{\overset{CH_3}{N^+}}-CH_2CH_2O)_nX$$

wherein X is a nonionic group selected from the group consisting of H, C_1-C_4 alkyl or hydroxyalkyl ester or ether groups, and mixtures thereof, a is from 0 to 20, preferably from 0 to 4 (e.g. ethylene, propylene, hexamethylene) b is 1 or 0; for cationic monoamines (b=0), n is at least 16, with a typical range of from 20 to 35; for cationic diamines (b=1), n is at least about 12 with a typical range of from about 12 to about 42.

Other dispersants/ anti-redeposition agents for use herein are described in EP-B-011965 and US 4,659,802 and US 4,664,848.

Suds Suppressing System

The detergent composition preferably comprises a suds suppresser at a level less than 5%, preferably 0.01% to 5%, preferably from 0.05% to 4%, most preferably from 0.1% to 3%, by weight of the composition Preferably the suds suppresser is either a soap, paraffin, wax, or any combination thereof. If the suds suppresser is a suds suppressing silicone, then the detergent composition preferably comprises less than 0.06% by weight extractable suds suppressing silicone.

Suitable suds suppressing systems for use herein may comprise essentially any known antifoam compound, including, for example silicone antifoam compounds and 2-alkyl alcanol antifoam compounds.

By antifoam compound it is meant herein any compound or mixtures of compounds which act such as to depress the foaming or sudsing produced by a solution of a detergent composition, particularly in the presence of agitation of that solution.

Particularly preferred antifoam compounds for use herein are silicone antifoam compounds defined herein as any antifoam compound including a silicone component. Such silicone antifoam compounds also typically contain a silica component. The term "silicone" as used herein, and in general throughout the industry, encompasses a variety of relatively high molecular weight polymers containing siloxane units and hydrocarbyl group of various types. Preferred silicone antifoam compounds are the siloxanes, particularly the polydimethylsiloxanes having trimethylsilyl end blocking units. Preferably the composition comprises less than 0.6% by weight extractable suds suppressing silicone.

Other suitable antifoam compounds include the monocarboxylic fatty acids and soluble salts thereof. These materials are described in US Patent 2,954,347, issued September 27, 1960 to Wayne St. John. The monocarboxylic fatty acids, and salts thereof, for use as suds suppresser typically have hydrocarbyl chains of 10 to 24 carbon atoms, preferably 12 to 18 carbon atoms. Suitable salts include the alkali metal salts such as sodium, potassium, and lithium salts, and ammonium and alkanolammonium salts.

Other suitable antifoam compounds include, for example, high molecular weight fatty esters (e.g. fatty acid triglycerides), fatty acid esters of monovalent alcohols, aliphatic C_{18}-C_{40} ketones (e.g. stearone) N-alkylated amino triazines such as tri- to hexa-alkylmelamines or di- to tetra alkyldiamine chlortriazines formed as products of cyanuric chloride with two or three moles of a primary or secondary amine containing 1 to 24 carbon atoms, propylene oxide, bis stearic acid amide and monostearyl di-alkali metal (e.g. sodium, potassium, lithium) phosphates and phosphate esters.

A preferred suds suppressing system comprises:

(a) antifoam compound, preferably silicone antifoam compound, most preferably a silicone antifoam compound comprising in combination:

  (i) polydimethyl siloxane, at a level of from 50% to 99%, preferably 75% to 95% by weight of the silicone antifoam compound; and

  (ii) silica, at a level of from 1% to 50%, preferably 5% to 25% by weight of the antifoam compound;

wherein said silica/silicone antifoam compound is incorporated at a level of less than 5%, preferably 0.01% to 5%, more preferably 0.05% to 4%, even more preferably 0.1% to 3%, by weight;

(b) a dispersant compound, most preferably comprising a silicone glycol rake copolymer with a polyoxyalkylene content of 72-78% and an ethylene oxide to propylene oxide ratio of from 1:0.9 to 1:1.1, at a level of less than 5%, preferably 0.01% to 5%, more preferably 0.05% to 4%, even more preferably 0.1% to 3%, by weight; a particularly preferred silicone glycol rake copolymer of this type is DCO544, commercially available from DOW Corning under the tradename DCO544;

(c) an inert carrier fluid compound, most preferably comprising a C_{16}-C_{18} ethoxylated alcohol with a degree of ethoxylation of from 5 to 50, preferably 8 to 15, at a level of less than 5%, preferably 0.01% to 5%, more preferably 0.05% to 4%, even more preferably 0.1% to 3%, by weight;

A highly preferred particulate suds suppressing system is described in EP-A-0210731 and comprises a silicone antifoam compound and an organic carrier material having a melting point in the range 50°C to 85°C, wherein the organic carrier material comprises a monoester of glycerol and a fatty acid having a carbon chain containing from 12 to 20 carbon atoms. EP-A-0210721 discloses other preferred particulate suds suppressing systems wherein the organic carrier material is a fatty acid or alcohol having a carbon chain containing from 12 to 20 carbon atoms, or a mixture thereof, with a melting point of from 45°C to 80°C.

Clay Softening System

The detergent composition of the invention may comprise a clay softening system comprising a clay mineral compound and optionally a clay flocculating agent.

The clay mineral compound is preferably a smectite clay compound. Smectite clays are disclosed in the US Patents No.s 3,862,058, 3,948,790, 3,954,632 and 4,062,647. European Patents No.s EP-A-299,575 and EP-A-313,146 in the name of the Procter and Gamble Company describe suitable organic polymeric clay flocculating agents.

Polymeric Dye Transfer Inhibiting Agents

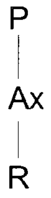
The detergent composition of the invention may also comprise from 0.01% to 10 %, preferably from 0.05% to 0.5% by weight of polymeric dye transfer inhibiting agents.

The polymeric dye transfer inhibiting agents are preferably selected from polyamine N-oxide polymers, copolymers of N-vinylpyrrolidone and N-vinylimidazole, polyvinylpyrrolidonepolymers or combinations thereof.

a) Polyamine N-oxide polymers

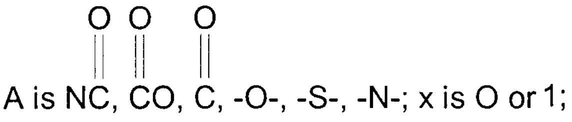
Polyamine N-oxide polymers suitable for use herein contain units having the following structure formula :

(I)



$$\begin{array}{c} P \\ | \\ Ax \\ | \\ R \end{array}$$

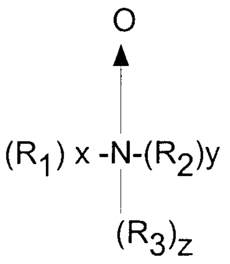
wherein P is a polymerisable unit, and



A is $N\overset{\overset{O}{\|}}{C}$, $\overset{\overset{O}{\|}}{C}O$, $\overset{\overset{O}{\|}}{C}$, -O-, -S-, -N-; x is O or 1;

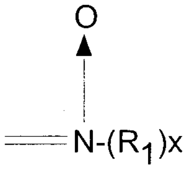
R are aliphatic, ethoxylated aliphatics, aromatic, heterocyclic or alicyclic groups or any combination thereof whereto the nitrogen of the N-O group can be attached or wherein the nitrogen of the N-O group is part of these groups.

The N-O group can be represented by the following general structures :



$$(R_1)\,x\,\text{-}\overset{\overset{O}{\uparrow}}{\underset{\underset{(R_3)_z}{|}}{N}}\text{-}(R_2)y$$

or



$$=\overset{\overset{O}{\uparrow}}{N}\text{-}(R_1)x$$

wherein R1, R2, and R3 are aliphatic groups, aromatic, heterocyclic or alicyclic groups or combinations thereof, x or/and y or/and z is 0 or 1 and wherein the nitrogen of the N-O group can be attached or wherein the nitrogen of the N-O group forms part of these groups. The N-O group can be part of the polymerisable unit (P) or can be attached to the polymeric backbone or a combination of both.

Suitable polyamine N-oxides wherein the N-O group forms part of the polymerisable unit comprise polyamine N-oxides wherein R is selected from aliphatic, aromatic, alicyclic or heterocyclic groups. One class of said polyamine N-oxides comprises the group of polyamine N-oxides wherein the nitrogen of the N-O group forms part of the R-group. Preferred polyamine N-oxides are those wherein R is a heterocyclic group such as pyrridine, pyrrole, imidazole, pyrrolidine, piperidine, quinoline, acridine and derivatives thereof.

Other suitable polyamine N-oxides are the polyamine oxides whereto the N-O group is attached to the polymerisable unit. A preferred class of these polyamine N-oxides comprises the polyamine N-oxides having the general formula (I) wherein R is an aromatic,heterocyclic or alicyclic groups wherein the nitrogen of the N-O functional group is part of said R group. Examples of these classes are polyamine oxides wherein R is a heterocyclic compound such as pyrridine, pyrrole, imidazole and derivatives thereof.

The polyamine N-oxides can be obtained in almost any degree of polymerisation. The degree of polymerisation is not critical provided the material has the desired water-solubility and dye-suspending power. Typically, the average molecular weight is within the range of 500 to 1000,000.

b) Copolymers of N-vinylpyrrolidone and N-vinylimidazole

Suitable herein are coploymers of N-vinylimidazole and N-vinylpyrrolidone having an average molecular weight range of from 5,000 to 50,000. The preferred copolymers have a molar ratio of N-vinylimidazole to N-vinylpyrrolidone from 1 to 0.2.

c) Polyvinylpyrrolidone

The detergent composition of the invention may also utilize polyvinylpyrrolidone ("PVP") having an average molecular weight of from 2,500 to 400,000. Suitable polyvinylpyrrolidones are commercially vailable from ISP Corporation, New York, NY and Montreal, Canada under the product names PVP K-15 (viscosity molecular weight of 10,000), PVP K-30 (average molecular weight of 40,000), PVP K-60 (average molecular weight of 160,000), and PVP K-90 (average molecular weight of 360,000). PVP K-15 is also available from ISP Corporation. Other suitable polyvinylpyrrolidones which are commercially available from BASF Cooperation include Sokalan HP 165 and Sokalan HP 12.

d) Polyvinyloxazolidone

The detergent composition of the invention may also utilize polyvinyloxazolidones as polymeric dye transfer inhibiting agents. Said polyvinyloxazolidones have an average molecular weight of from 2,500 to 400,000.

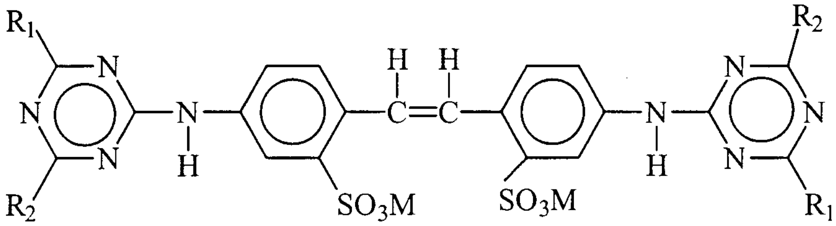
e) Polyvinylimidazole

The detergent composition of the invention may also utilize polyvinylimidazole as polymeric dye transfer inhibiting agent. Said polyvinylimidazoles preferably have an average molecular weight of from 2,500 to 400,000.

Optical Brightener

The detergent composition of the invention may also optionally comprise from 0.005% to 5% by weight of certain types of hydrophilic optical brighteners.

Hydrophilic optical brighteners useful herein include those having the structural formula:



wherein R_1 is selected from anilino, N-2-bis-hydroxyethyl and NH-2-hydroxyethyl; R_2 is selected from N-2-bis-hydroxyethyl, N-2-hydroxyethyl-N-methylamino, morphilino, chloro and amino; and M is a salt-forming cation such as sodium or potassium.

When in the above formula, R_1 is anilino, R_2 is N-2-bis-hydroxyethyl and M is a cation such as sodium, the brightener is 4,4',-bis[(4-anilino-6-(N-2-bis-hydroxyethyl)-s-triazine-2-yl)amino]-2,2'-stilbenedisulfonic acid and disodium salt. This particular brightener species is commercially marketed under the tradename Tinopal-UNPA-GX by Ciba-Geigy Corporation. Tinopal-UNPA-GX is the preferred hydrophilic optical brightener useful in the detergent compositions herein.

When in the above formula, R_1 is anilino, R_2 is N-2-hydroxyethyl-N-2-methylamino and

M is a cation such as sodium, the brightener is 4,4'-bis[(4-anilino-6-(N-2-hydroxyethyl-N-methylamino)-s-triazine-2-yl)amino]2,2'-stilbenedisulfonic acid disodium salt. This particular brightener species is commercially marketed under the tradename Tinopal 5BM-GX by Ciba-Geigy Corporation.

When in the above formula, R_1 is anilino, R_2 is morphilino and M is a cation such as sodium, the brightener is 4,4'-bis[(4-anilino-6-morphilino-s-triazine-2-yl)amino]2,2'-stilbenedisulfonic acid, sodium salt. This particular brightener species is commercially marketed under the tradename Tinopal AMS-GX by Ciba Geigy Corporation.

Cationic Fabric Softening Agents

Cationic fabric softening agents can also be incorporated into the detergent composition of the invention or in compositions containing the detergent component in accordance with the present invention. Suitable cationic fabric softening agents include the water insoluble tertiary amines or dilong chain amide materials as disclosed in GB-A-1 514 276 and EP-B-0 011 340.

Cationic fabric softening agents are typically incorporated at total levels of from 0.5% to 15% by weight, normally from 1% to 5% by weight.

Other Optional Ingredients

Other optional detergent ingredients suitable for inclusion in the compositions or detergent component of the invention include highly preferably perfumes, colours and filler salts, with sodium sulphate being a preferred filler salt.

Form of Composition

The detergent composition of the invention can be made by a variety of methods, including dry-mixing, extruding, compacting and agglomerating of the various compounds comprised in the detergent composition. The particles can be present in the compositions as a separate component of the composition, or can be added to other components or compounds of the composition. The granular detergent composition of the invention can take a variety of physical forms including granular, flakes or extrudate.

Laundry Washing Method

Laundry methods herein typically comprise treating soiled laundry with an aqueous wash solution for washing by machine or for washing by hand, having dissolved or dispensed therein an effective amount of a laundry composition. By an effective amount of the detergent composition it is meant from 40g to 300g of product dissolved or dispersed in a wash solution of volume from 1 to 65 litres, as are typical product dosages and wash solution volumes commonly employed in conventional laundry methods. Other laundry washing processes known in the art can also be used.

Packaging for the Compositions

Commercially marketed executions of the bleaching compositions can be packaged in any suitable container including those constructed from paper, cardboard, plastic materials and any suitable laminates. A preferred packaging execution is described in European Application No. 94921505.7.

Examples

Abbreviations used in examples

In the detergent compositions, the abbreviated component identifications have the following meaning:

LAS	:	Sodium linear C11-13 alkyl benzene sulfonate
LAS (I)	:	Potassium linear or branched C11-13 alkyl benzene sulfonate
TAS	:	Sodium tallow alkyl sulfate
CxyAS	:	Sodium C1x - C1y alkyl sulfate
C46SAS	:	Sodium C14 - C16 secondary (2,3) alkyl sulfate
CxyEzS	:	Sodium C1x-C1y alkyl sulfate condensed with z moles of ethylene oxide
CxyEz	:	C1x-C1y predominantly linear primary alcohol condensed with an average of z moles of ethylene oxide
QAS	:	R2.N+(CH3)2(C2H4OH) with R2 = C12 - C14
QAS 1	:	R2.N+(CH3)2(C2H4OH) with R2 = C8 - C 11
APA	:	C8 - C10 amido propyl dimethyl amine
Soap	:	Sodium linear alkyl carboxylate derived from an 80/20 mixture of tallow and coconut fatty acids
STS	:	Sodium toluene sulphonate
CFAA	:	C12-C14 (coco) alkyl N-methyl glucamide
TFAA	:	C16-C18 alkyl N-methyl glucamide
TPKFA	:	C12-C14 topped whole cut fatty acids
STPP	:	Anhydrous sodium tripolyphosphate
TSPP	:	Tetrasodium pyrophosphate
Zeolite A	:	Hydrated sodium aluminosilicate of formula Na12(A1O2SiO2)12.27H2O having a primary particle size in the range from 0.1 to 10 micrometers (weight expressed on an anhydrous basis)

NaSKS-6 : Crystalline layered silicate of formula d- Na2Si2O5

Citric acid I : Anhydrous citric acid, 80% having a particle size of from 40 microns to 70 microns, and having a volume median particle size of 55 microns

Citric acid II : Anhydrous or monohydrate citric acid, 80% having a particle size of from 15 microns to 40 microns, having a volume average particle size of 25 microns

Malic acid : Anhydrous malic acid, 80% having a particle size of from 50 microns to 100 microns, having a volume median particle size of 75 microns

Maleic acid : Anhydrous maleic acid, 80% having a particle size of from 5 microns to 30 microns, having a volume median particle size of 15 microns

Tartaric acid : Anhydrous tartaric acid, 80% having a particle size of from 25 microns to 75 microns, having a volume median particle size of 50 microns

Carbonate I : Anydrous sodium carbonate having 80% by volume of particles with a particle size from 50 microns to 150 microns with a volume median particle size of 100 microns

Carbonate II : Anydrous sodium carbonate having 80% by volume of particles with a particle size from 35 microns to 75 microns, having a volume median particle size of 55 microns

Bicarbonate II: Anhydrous sodium bicarbonate having 80% by volume of particles with a particle size from 100 microns to 200 microns with a volume median particle size of 150 microns

Bicarbonate I : Anydrous sodium bicarbonate having 80% by volume of particles with a particle size from 15 microns to 40 microns, having a volume median particle size of 25 microns

Silicate : Amorphous sodium silicate (SiO2:Na2O = 2.0:1)

Sulfate : Anhydrous sodium sulfate

Mg sulfate : Anhydrous magnesium sulfate

Citrate	Tri-sodium citrate dihydrate of activity 86.4% with a particle size distribution between 425µm and 850µm
MA/AA	Copolymer of 1:4 maleic/acrylic acid, average molecular weight about 70,000
MA/AA (1)	Copolymer of 4:6 maleic/acrylic acid, average molecular weight about 10,000
AA	Sodium polyacrylate polymer of average molecular weight 4,500
CMC	Sodium carboxymethyl cellulose
Cellulose ether	Methyl cellulose ether with a degree of polymerization of 650 available from Shin Etsu Chemicals
Protease	Proteolytic enzyme, having 3.3% by weight of active enzyme, sold by NOVO Industries A/S under the tradename Savinase
Protease I	Proteolytic enzyme, having 4% by weight of active enzyme, as described in WO 95/10591, sold by Genencor Int. Inc.
Alcalase	Proteolytic enzyme, having 5.3% by weight of active enzyme, sold by NOVO Industries A/S
Cellulase	Cellulytic enzyme, having 0.23% by weight of active enzyme, sold by NOVO Industries A/S under the tradename Carezyme
Amylase	Amylolytic enzyme, having 1.6% by weight of active enzyme, sold by NOVO Industries A/S under the tradename Termamyl 120T
Lipase	Lipolytic enzyme, having 2.0% by weight of active enzyme, sold by NOVO Industries A/S under the tradename Lipolase
Lipase (1)	Lipolytic enzyme, having 2.0% by weight of active enzyme, sold by NOVO Industries A/S under the tradename Lipolase Ultra
Endolase	Endoglucanase enzyme, having 1.5% by weight of active enzyme, sold by NOVO Industries A/S
PB4	Particle containing sodium perborate tetrahydrate of nominal formula NaBO2.3H2 O, the particles having a weight average particle size of 950 microns, 85% particles having a particle size of from 850 microns to 950 microns
PB1	Particle containing anhydrous sodium perborate bleach of nominal

		formula NaBO2.H 2O2, the particles having a weight average particle size of 800 microns, 85% particles having a particle size of from 750 microns to 950 microns
Percarbonate	:	The sodium percarbonate source of the invention as described hereinbefore (see detailed description of the invention, percarbonate source)
Coated percarbonate:		The coated sodium percarbonate source of the invention, as described hereinbefore (see detailed description of the invention, percarbonate source), as described hereinafter, with 4.5% by weight coating comprising sodium bicarbonate and/or sodium sulphate and prepared by a fluidised bed process (percarbonate sample 2) or with 4.5% by weight sodium carbonate and/or sodium sulphate and prepared by a crystalline/classification process, ex Solvay
NOBS	:	Particle comprising nonanoyloxybenzene sulfonate in the form of the sodium salt, the particles having a weight average particle size of 750 microns to 900 microns
NAC-OBS	:	Particle comprising (6-nonamidocaproyl) oxybenzene sulfonate, the particles having a weight average particle size of from 825 microns to 875 microns
TAED I	:	Particle containing tetraacetylethylenediamine, the particles having a weight average particle size of from 700 microns to 1000 microns
TAED II	:	Tetraacetylethylenediamine of a particle size from 150 microns to 600 microns
DTPA	:	Diethylene triamine pentaacetic acid
DTPMP	:	Diethylene triamine penta (methylene phosphonate), marketed by Monsanto under the Tradename Dequest 2060
Photoactivated	:	Sulfonated zinc phthlocyanine encapsulated in bleach (1) dextrin soluble polymer

Photoactivated	:	Sulfonated alumino phthlocyanine encapsulated in bleach (2) dextrin soluble polymer
Brightener 1	:	Disodium 4,4'-bis(2-sulphostyryl)biphenyl
Brightener 2	:	Disodium 4,4'-bis(4-anilino-6-morpholino-1.3.5-triazin-2-yl)amino) stilbene-2:2'-disulfonate
EDDS	:	Ethylenediamine-N,N'-disuccinic acid, (S,S) isomer in the form of its sodium salt.
HEDP	:	1,1-hydroxyethane diphosphonic acid
PEGx	:	Polyethylene glycol, with a molecular weight of x (typically 4,000)
PEO	:	Polyethylene oxide, with an average molecular weight of 50,000
TEPAE	:	Tetraethylenepentaamine ethoxylate
PVI	:	Polyvinyl imidosole, with an average molecular weight of 20,000
PVP	:	Polyvinylpyrolidone polymer, with an average molecular weight of 60,000
PVNO	:	Polyvinylpyridine N-oxide polymer, with an average molecular weight of 50,000
PVPVI	:	Copolymer of polyvinylpyrolidone and vinylimidazole, with an average molecular weight of 20,000
QEA	:	bis((C2H5O)(C2H4O)n)(CH3) -N+-C6H12-N+-(CH3) bis((C2H5O)-(C2H4 O))n, wherein n = from 20 to 30
SRP 1	:	Anionically end capped poly esters
SRP 2	:	Diethoxylated poly (1, 2 propylene terephtalate) short block polymer
PEI	:	Polyethyleneimine with an average molecular weight of 1800 and an average ethoxylation degree of 7 ethyleneoxy residues per nitrogen
Silicone antifoam	:	Polydimethylsiloxane foam controller with siloxane-oxyalkylene copolymer as dispersing agent with a ratio of said foam controller to said dispersing agent of 10:1 to 100:1
Opacifier	:	Water based monostyrene latex mixture, sold by BASF Aktiengesellschaft under the tradename Lytron 621

Wax : Paraffin wax

Example 1

The mean particle size of the detergent composition was determined by the method described hereinbefore (see detailed description of the invention, mean particle size).

A detergent composition used to determine the mean particle size (MPS) is described hereinbefore (see detailed description of the invention, mean particle size). The mean particle size of a detergent composition comprising 10% by weight sodium perborate monohydrate (sodium perborate sample) is also shown, along with two detergent compositions comprising 10% by weight sodium percarbonate (sodium percarbonate sample 1 and 2, see below for a more detailed description) before (0h) and after exposure for 24 hours (24h) to conditions of 32°C and 80% relative humidity by the method described hereinbefore (see detailed description of the invention, mean particle size).

The sodium percarbonate sample 1 comprises a coated sodium percarbonate comprising or with 4.5% by weight sodium carbonate and/or sodium sulphate and prepared by a crystalline/classification process, ex Solvay.

The sodium percarbonate sample 2 comprises a coated sodium percarbonate comprising 4.5% by weight a coating comprising sodium bicarbonate and/or sodium sulphate and the coated sodium percarbonate is prepared by a fluidised bed process

Results

The particle size distribution(PSD)s of the said detergent compositions are shown below.

Particle Size	**Sodium perborate sample**		**Sodium percarbonate sample 1**	
(Sieve size)	**PSD (0h)**	**PSD (24h)**	**PSD (0h)**	**PSD (24h)**
2000	0	0.16	0.02	0.58
180	2.95	10.89	5.19	7.23
850	6.71	18.84	11.13	14.01
600	11.12	21.29	16.3	18.56
425	16.88	20.16	15.83	25.02
250	37.32	30.85	29.54	34.14
Pan	44.82	26.24	41.95	26.2
MPS (µm)	**311**	**473**	**346**	**437**

Particle Size	**Sodium percarbonate sample 2**	
(Sieve size)	**PSD (0h)**	**PSD (24h)**
2000	0	0.01
180	4.45	3.98
850	9.31	9.52
600	19.6	20.92
425	16.75	21.73
250	28.62	37.89
Pan	41.02	32.67
MPS (µm)	**351**	**386**

	Perborate sample		**Percarbonate sample 1**		**Percarbonate sample 2**	
Time	**0h**	**24h**	**0h**	**24h**	**0h**	**24h**
MPS (µm)	387	593	456	540	452	467
Increase in MPS (µm)		206		84		5
Increase in MPS (% of original MPS)		**52%**		**26%**		**10%**

These data show that when selected sodium percarbonate, as opposed to sodium perborate is comprised by a detergent composition, the detergent composition has, after being exposed simultaneously to 32°C and 80% relative humidity for 24 hours by the method described hereinbefore (see detailed description of the invention, mean particle size), a mean particle size of 100% to 130% of the original mean particle size.

Example 2

	Perborate sample		**Percarbonate sample 1**		**Percarbonate sample 2**	
	wk0	**wk8**	**wk0**	**wk8**	**wk0**	**wk8**
Bulk density (g/l)	485	423	475	436	475	446
Bulk density (% of original)	100	**87**	100	**92**	100	**94**

These data show that when selected sodium percarbonate, as opposed to sodium perborate is comprised by a detergent composition, the detergent composition has, after being stored in conditions of 32°C and 80% relative humidity for 24 hours by the method described hereinbefore (see detailed description of the invention, mean particle size), a bulk density of 90% to 100% of the original mean particle size.

Example 3

Table I

The following compositions are in accordance with the invention.

	A	B	C	D	E	F	G	H	I
TPKFA	5			2			3		1

LAS	3	20	1	15	20				2
QAS							2		10
C28E3S						3		2	5
C28E7			10	2			1	5	10
C68AS						20	15	10	
TAS	15								
MES				1		6	4		15
SADS			10			3		1	
STPP	45	20	25	10	30	1		25	
Zeolite A		5		10	1	6	16		15
Silicate	1	10	6	9	4	15	6	4	6
Carbonate		20	10	15	5		20	5	
Dried carbonate	1						1	4	10
DTPA	0.4	1	0.3	2	0.1			2	
Mg Sulfate		0.2				2	0.1		
Citric Acid	6		5	2.5		3	8	4	0.1
MA/AA		0.8	0.9		1		0.2		
MA/AA (1)	0.1			0.8					
AA						1		0.7	
CMC			0.2	0.4	0.4		0.8		
SRP1		0.1			0.2				
SRP2			0.2					0.1	0.2
Protease			0.1			0.6			0.8
Protease I			0.2		0.1		0.6		
Cellulase	0.1			0.4				0.2	
Amylase		0.6				0.1	0.05		
Amylase II			0.2		0.6				0.1
Lipase						0.1	0.6		0.5
Lipase I				0.1				0.2	
Endolase	0.6	0.2							

PB1	1								
NOBS	2	1	0.5	4	1.5	4	2	1.5	3
TAED	1	0.5	2	0.6	2	1.6		1	1
Percarbonate	3	4	12	1	4	1	2	3	1
Coated percarbonate	12	12	3	4	7	4	6	7	5
Photoactivated bleach 1			0.05			0.05			
Photoactivated bleach 2				0.05					
Brightener 1		0.3			0.05		0.1		
Brightener 2			0.1		0.05				0.3
Silicone antifoam		0.2		0.1		0.05		0.05	
Perfume	0.5				0.2		1		2
Misc/minor	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%

Table II

The following compositions are in accordance with the invention.

	J	K	L	M	N	O	P	Q	R
TPKFA			2						
LAS	20	15	3		20	15	15	20	15
QAS		2	0.5						
C28E3S				4					
C28E7	2	1	20						
C68AS				10					
TAS				6			5		
MES					8				
SADS				3					
STPP	35	25	25	15	20	25	30	10	15
Zeolite A			8	20				4	

Silicate	8	7	1.4	6	1.5	9		9	9
Carbonate	10	12	20	9	12	20	10	15	15
Dried carbonate		1	3	7					
DTPA	0.9	0.5	1	1	0.3	0.5	0.4	0.8	0.9
Mg Sulfate	0.6	1.4			0.8		1.4	1.2	1
Citric Acid		4		10					
MA/AA	1	0.5		0.5	1	0.5	1	1.5	0.1
MA/AA (1)			0.6						0.75
AA				1		0.5			
CMC	0.5	0.4	0.8	1	0.4	0.2	0.4	0.4	0.3
SRP1	0.3			0.1					
SRP2			0.4	0.1					
Protease	0.7	0.4	0.1	0.2	0.2	0.2	0.1		
Protease I		0.2	0.1	0.05	0.4	0.3	0.6	0.1	0.7
Cellulase		0.1	0.2	0.05					
Amylase		0.1		0.1			0.6	0.6	
Amylase II		0.05	0.1		0.15	0.3			
Lipase		0.2		0.1					
Lipase I				0.1					
Endolase									
PB1									
NOBS	1.9	1.5	2	0.5	1.5	1.9		1	0.7
TAED			0.5	1.5			0.6		
Percarbonate	3	4		2			2		4
Coated percarbonate	4	10	5	10	7	8	6	10	4
Photoactivated bleach 1		0.01							
Photoactivated bleach 2			0.01						
Brightener 1	0.05	0.05	0.05	0.05	0.05	0.1	0.05		
Brightener 2	0.1		0.1		0.05			0.1	0.05
Silicone antifoam	0.2	0.15		0.05				0.01	0.01

Perfume	0.25	0.4	0.3	0.1	0.6	0.4	0.2	0.4	
Misc/minor	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%

What is claimed is:

1. A granular bleach-containing detergent composition with a bulk density of less than 570g/l, characterised in that the detergent composition comprises a percarbonate source, where the percarbonate source comprises a salt of percarbonate, and that, after being exposed simultaneously to 32°C and 80% relative humidity for 24 hours, the detergent composition has a mean particle size of 100% to 130% of the original mean particle size.

2. A detergent composition according to claim 1, characterised in that, after being stored in conditions of 32°C and 80% relative humidity for 8 weeks, the detergent composition has a bulk density of 90 to 100% of the original bulk density.

3. A detergent composition according to any preceding claim, characterised in that, after being exposed simultaneously to 32°C and 80% relative humidity for 24 hours, the detergent composition has a mean particle of 100% to 120%, preferably 100% to 110%, of the original mean particle size.

4. A granular bleach-containing detergent composition with a bulk density of less than 570g/l comprising a percarbonate source and less than 0.06% by weight extractable suds suppressing silicone, where the percarbonate source comprises a salt of percarbonate comprising less than 2% by weight chloride ions, or chloride-containing compounds.

5. A detergent composition according to any preceding claim, whereby the percarbonate source is prepared by a fluidised bed process.

6. A detergent composition according to any preceding claim, whereby the percarbonate source comprises less than 2% by weight chloride ions.

7. A detergent composition according to any preceding claim, whereby the detergent

composition comprises less than 36%, preferably from 1% to 35%, more preferably from 10% to 35%, even more preferably 12% to 30%, by weight an alkali or earth alkali metal salt of phosphate.

8. A detergent composition according to any preceding claim, whereby the detergent composition comprises less than 5% by weight a suds suppresser, where the suds suppresser is either a soap, paraffin, wax, or any combination thereof.

9. A detergent composition according to any preceding claim, whereby the detergent composition comprises less than 0.06% by weight extractable suds suppressing silicone.

10. A detergent composition according to any preceding claim, whereby the detergent composition has a moisture content of less than 10% by weight.

11. A detergent composition according to any preceding claim, whereby the detergent composition has a reserve alkalinity greater than 6g NaOH per 100g detergent composition, measured to a pH of 7.5.

12. A detergent composition according to any preceding claim, whereby the percarbonate source is present at a level from 0.01% to 12%, preferably from 2% to 10%, more preferably from 3% to 7%, by weight.

13. A detergent composition according to any preceding claim, whereby the detergent composition comprises 0.05% to 2% by weight a metal chelating agent.

14. A detergent composition according to any preceding claim, whereby the detergent composition comprises a coated salt of percarbonate particle, characterised in that the coating comprises one or more alkali metal salt, preferably sodium carbonate, sodium bicarbonate, sodium silicate, sodium sulphate, or any combination thereof.

15. A detergent composition according to claim 13, whereby the coated salt of percarbonate particle comprises a coating present at a level of 0.01 % to 20%, preferably 4 to 15%, more preferably 4% to 10%, by weight of the coated salt of percarbonate particle.

INTERNATIONAL SEARCH REPORT

Inte onal Application No
PCT/US 00/30870

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C11D17/00 C11D3/39 C11D3/10 C11D17/06

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 C01B 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)

PAJ, EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 902 682 A (BEWERSDORF MARTIN ET AL) 11 May 1999 (1999-05-11) claims; examples; tables	1-6, 8-12,14, 15
X	GB 1 466 799 A (INTEROX) 9 March 1977 (1977-03-09) claims 1-18; tables 1-5	1-12,14, 15
X	GB 1 538 893 A (INTEROX) 24 January 1979 (1979-01-24) page 2, line 16 - line 17; claims 1-23; examples; tables	1-12,14, 15
A	EP 0 623 553 A (MITSUBISHI GAS CHEMICAL CO) 9 November 1994 (1994-11-09) page 4, line 40 - line 46; examples 3,5	1-15

-/--

☒ 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

"E" earlier document but published on or after the international filing date

"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

Date of the actual completion of the international search 28 February 2001	Date of mailing of the international search report 07/03/2001
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer Loiselet-Taisne, S

1

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Inte onal Application No
PCT/US 00/30870

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
P,X	EP 0 970 917 A (DEGUSSA) 12 January 2000 (2000-01-12) claims 1-4; examples 1-6 ---	1-12,14, 15
X	US 3 951 838 A (YATES PAUL CLIFFORD ET AL) 20 April 1976 (1976-04-20) claims 1-10; example 1; table I -----	1-12,14, 15

1

INTERNATIONAL SEARCH REPORT

Information on patent family members

Inte onal Application No
PCT/US 00/30870

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5902682 A	11-05-1999	DE 4324104 A	19-01-1995
		AT 162497 T	15-02-1998
		AU 6647994 A	13-02-1995
		CA 2166281 A	26-01-1995
		CN 1127498 A	24-07-1996
		CZ 9600018 A	15-05-1996
		DE 59405122 D	26-02-1998
		WO 9502555 A	26-01-1995
		EP 0710215 A	08-05-1996
		ES 2113102 T	16-04-1998
		FI 960208 A	16-01-1996
		HU 72833 A	28-05-1996
		IL 110331 A	04-01-1998
		JP 9500084 T	07-01-1997
		PL 312608 A	29-04-1996
		SK 4296 A	08-05-1996
		TR 27995 A	16-11-1995
		ZA 9405218 A	27-02-1995
GB 1466799 A	09-03-1977	LU 67482 A	21-11-1974
		LU 68831 A	20-08-1975
		AU 6762374 A	09-10-1975
		BE 813645 A	14-10-1974
		BR 7403188 A	30-12-1975
		CH 586160 A	31-03-1977
		DE 2417572 A	14-11-1974
		ES 425334 A	01-06-1976
		FR 2226460 A	15-11-1974
		IT 1009862 B	20-12-1976
		JP 1207098 C	11-05-1984
		JP 50121174 A	22-09-1975
		JP 58024361 B	20-05-1983
		SE 414646 B	11-08-1980
		US 4105827 A	08-08-1978
		ZA 7402114 A	26-03-1975
GB 1538893 A	24-01-1979	LU 72575 A	10-02-1977
		BE 842014 A	22-11-1976
		DE 2622610 A	09-12-1976
		FR 2324574 A	15-04-1977
		IT 1063355 B	11-02-1985
EP 0623553 A	09-11-1994	DE 69404543 D	04-09-1997
		DE 69404543 T	04-12-1997
		JP 7017710 A	20-01-1995
		US 5462804 A	31-10-1995
		JP 7017703 A	20-01-1995
		JP 7069606 A	14-03-1995
EP 0970917 A	12-01-2000	DE 19830946 A	13-01-2000
		BR 9902669 A	08-03-2000
		SK 93099 A	16-05-2000
		TR 9901597 A	21-02-2000
US 3951838 A	20-04-1976	DE 2448453 A	24-04-1975
		BE 820741 A	04-04-1975
		CA 1032333 A	06-06-1978
		ES 430855 A	01-10-1976

INTERNATIONAL SEARCH REPORT

Information on patent family members

Inte onal Application No
PCT/US 00/30870

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 3951838 A		FR 2247533 A	09-05-1975
		GB 1439909 A	16-06-1976
		IT 1022733 B	20-04-1978
		JP 50065500 A	03-06-1975
		SE 7412635 A	11-04-1975

Form PCT/ISA/210 (patent family annex) (July 1992)