

(19)



Europäisches Patentamt

European Patent Office

Office européen des brevets



(11)

EP 0 790 297 A1

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication:
20.08.1997 Bulletin 1997/34

(51) Int. Cl.⁶: **C11D 3/39**, C11D 17/00,
C11D 1/10

(21) Application number: **96870012.0**

(22) Date of filing: **16.02.1996**

(84) Designated Contracting States:
AT BE CH DE DK ES FR GB GR IE IT LI LU NL PT SE

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(54) **Bleaching compositions**

(57) The present invention relates to liquid compositions comprising a peroxygen bleach, a liquid hydrophobic bleach activator and a long chain (C12-C16) acyl sarcosinate, being formulated either as an emulsion or a microemulsion. The present invention further encompasses a process for pretreating soiled fabrics with a liquid composition (pH=0 to 7) comprising a long chain (C12-C16) acyl sarcosinate, whereby improved stain removal performance is delivered.

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DescriptionTechnical field

5 The present invention relates to stable peroxygen bleach-containing compositions particularly suitable to be used as pretreater.

Background

10 Peroxygen bleach-containing compositions have been extensively described in laundry applications as laundry detergents, laundry additives or even laundry pretreaters.

Indeed, it is known to use liquid compositions comprising a peroxygen bleach and a bleach activator in laundry pre-treatment applications. Although said liquid bleaching compositions provide good bleaching performance when used to pretreat a soiled fabric, there is still some room to further improve them regarding for example the stains/soils removal performance delivered when pretreating said soiled fabric.

It is thus an object of the present invention to provide improved stain removal performance on a variety of stains/soils under pretreatment conditions, i.e., when applied directly onto the soiled fabrics, and left to act onto said fabrics before washing said fabrics. More particularly, it is an object of the present invention to provide liquid compositions containing a peroxygen bleach and a liquid hydrophobic bleach activator, which deliver improved stain removal performance on a variety of stains/soils under pretreatment conditions while not compromising on their bleaching performance.

It has now been found that the above object can be met by using a liquid composition (pH=0 to 7) comprising a long chain (C12-C16) acyl sarcosinate to pretreat soiled fabrics. Indeed, it has been found that the addition of a long chain (C12-C16) acyl sarcosinate in a liquid composition (pH=0 to 7), e.g., in a liquid composition comprising a peroxygen bleach and optionally a liquid hydrophobic bleach activator, allows to deliver improved stain removal performance on fabrics under pretreatment conditions, as compared to the stain removal performance delivered by the same composition without said long chain acyl sarcosinate or to the same composition but with another anionic surfactant like for instance C12 alkyl sulfate instead of said long chain acyl sarcosinate. Thus in its broadest aspect, the present invention encompasses the use of said long chain acyl sarcosinate, in a liquid composition (pH=0 to 7), for improving the stain removal performance of said composition under pretreatment conditions. The present invention also encompasses liquid compositions comprising said long chain acyl sarcosinate, a peroxygen-bleach and a liquid hydrophobic bleach activator. Indeed, it has further been found that the addition of said long chain acyl sarcosinates, in the liquid peroxygen bleach-containing compositions of the present invention comprising said liquid hydrophobic bleach activator, contributes to emulsify said hydrophobic bleach activator so that said compositions are formulated either as microemulsions or as emulsions. Thus, the compositions according to the present invention allow to provide improved stain removal performance when pretreating soiled fabrics while exhibiting also excellent chemical stability, even upon prolonged storage periods.

An advantage of the present invention is that the improved stain removal performance obtained when pretreating fabrics with the compositions of the present invention is noticeable on a variety of stains/soils including greasy/oil stains, like make-up, lipstick, dirty motor oil and mineral oil, greasy food like mayonnaise and spaghetti sauce, bleachable stains like tea, wine, grass and enzymatic stains like blood.

EP-A-677575 discloses liquid compositions formulated as emulsions comprising two phases, each phase comprising a nonionic surfactant, said compositions comprising a peroxygen bleach in one of the phases and a bleach activator in the other phase, said compositions further comprising an anionic surfactant. EP-A-677575 further discloses the use, in a liquid peroxygen bleach composition, of an anionic surfactant to reduce skin itching of the user when it has come in contact with said composition. Acyl sarcosinates are mentioned amongst the anionic surfactants but no specific acyl sarcosinates are disclosed therein, nor exemplified.

Co-pending European patent application number 95203330.6 discloses a liquid bleaching composition comprising hydrogen peroxide, a liquid hydrophobic bleach activator, said composition being formulated as a microemulsion of said hydrophobic liquid bleach activator in a matrix comprising water, said hydrogen peroxide, a hydrophilic surfactant system comprising a nonionic surfactant and an anionic surfactant. Acyl sarcosinates are mentioned amongst the anionic surfactants but no specific acyl sarcosinates are disclosed therein, nor exemplified.

WO 95/27035 discloses a fabric washing detergent composition comprising an organic surfactant system (2%-40%), a non-aqueous system (0.5%-55%), a water-soluble polymeric detergency builder (0.1%-5%) and water, wherein the surfactant system and the non-aqueous solvent together form a stable oil-in-water microemulsion. These compositions are useful as pretreatment products as well as main wash products. No acyl sarcosinates are disclosed, let alone long chain acyl sarcosinates. Also no peroxygen bleaches are disclosed.

WO 95/27033 and WO 95/27034 disclose particular microemulsions suitable to be used as pretreatment products. However, no acyl sarcosinates are disclosed, let alone long chain acyl sarcosinates. Also no peroxygen bleaches are

disclosed.

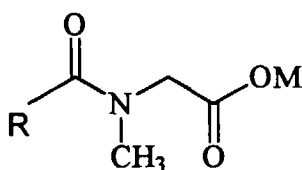
EP 318 470 discloses a laundry pre-treatment composition in stick form (solid form) which is suitable for pre-spotting onto stained fabric before laundering. Bleach like percarbonates and perborates are disclosed. No acyl sarcosinates are disclosed, let alone long chain acyl sarcosinates.

US 4613452 discloses emulsions containing an enol ester activator, hydrogen peroxide and nonionic and anionic emulsifiers. However, no acyl sarcosinates are disclosed, let alone long chain acyl sarcosinates.

EP 92 932 discloses emulsions comprising nonionic surfactants, a bleach, a bleach activator and, possibly anionic surfactants. However, no acyl sarcosinates are disclosed, let alone long chain acyl sarcosinates.

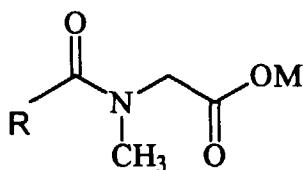
Summary of the invention

The present invention encompasses a liquid composition comprising a peroxygen bleach, a liquid hydrophobic bleach activator and a long chain acyl sarcosinate according to the formula



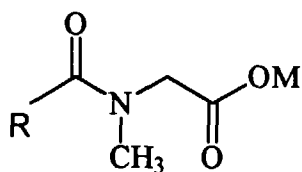
wherein M is hydrogen or a cationic moiety, and wherein R is an alkyl group of from 11 to 15 carbon atoms, said composition being formulated either as a microemulsion or as an emulsion.

The present invention also encompasses a process of pretreating soiled fabrics with a liquid composition having a pH up to 7 and comprising a long chain acyl sarcosinate according to the formula



wherein M is hydrogen or a cationic moiety, and wherein R is an alkyl group of from 11 to 15 carbon atoms, said process comprising the steps of applying said composition in its neat form onto the fabric and allowing said composition to remain in contact with said fabric, before said fabric is washed.

The present invention further encompasses the use of a long chain acyl sarcosinate according to the formula



wherein M is hydrogen or a cationic moiety, and wherein R is an alkyl group of from 11 to 15 carbon atoms, in a liquid composition having a pH up to 7, preferably in a liquid peroxygen bleach-containing composition, for improving the stain removal performance of said composition under pretreatment conditions, i.e., when pretreating a soiled fabric with said composition before said fabric is washed.

Detailed description of the invention

As an essential element the compositions of the present invention comprise a peroxygen bleach. Preferred peroxygen bleach is hydrogen peroxide, or a water soluble source thereof, or mixtures thereof. Hydrogen peroxide is most preferred to be used in the compositions of the present invention. Indeed, the presence of peroxygen bleach, preferably hydrogen peroxide, provides strong cleaning benefits which are particularly noticeable in laundry applications. As used

herein a hydrogen peroxide source refers to any compound which produces hydrogen peroxide when said compound is in contact with water.

Suitable water-soluble sources of hydrogen peroxide for use herein include percarbonates, persilicate, persulphate such as monopersulfate, perborates, peroxyacids such as diperoxydodecandioic acid (DPDA), magnesium perphthalic acid, perbenzoic and alkylperbenzoic acids, and mixtures thereof.

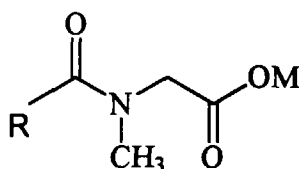
Typically, the compositions of the present invention comprise from 0.01% to 20% by weight of the total composition of said peroxygen bleach or mixtures thereof, preferably from 1% to 15%, and most preferably from 2% to 10%.

As another ingredient, the compositions of the present invention comprise a liquid hydrophobic bleach activator or mixtures thereof. By "bleach activator", it is meant herein a compound which reacts with hydrogen peroxide to form a peracid. The peracid thus formed constitutes the activated bleach. By "hydrophobic bleach activator", it is meant herein a bleach activator which is not substantially and stably miscible with water. Typically, such hydrophobic bleach activators have a secondary HLB of below 11, preferably below 10. Secondary HLB is known to those skilled in the art and is defined for example in "Emulsions theory and practice" by P. Becher, Reinhold, New York, 1957 or in "Emulsion science" by P. Sherman, Academic Press, London, 1969.

Suitable hydrophobic bleach activators to be used herein include those belonging to the class of esters, amides, imides, or anhydrides. Examples of suitable compounds of this type are disclosed in British Patent GB 1 586 769 and GB 2 143 231 and a method for their formation into a prilled form is described in European Published Patent Application EP-A-62 523. Suitable examples of such compounds to be used herein are tetracetyl ethylene diamine (TAED), sodium 3,5,5 trimethyl hexanoyloxybenzene sulphonate, diperoxy dodecanoic acid as described for instance in US 4 818 425 and nonylamide of peroxyadipic acid as described for instance in US 4 259 201 and n-nonanoyloxybenzenesulphonate (NOBS). Also suitable are N-acyl caprolactams selected from the group consisting of substituted or unsubstituted benzoyl caprolactam, octanoyl caprolactam, nonanoyl caprolactam, hexanoyl caprolactam, decanoyl caprolactam, undecenoyl caprolactam, formyl caprolactam, acetyl caprolactam, propanoyl caprolactam, butanoyl caprolactam pentanoyl caprolactam or mixtures thereof. A particular family of bleach activators of interest was disclosed in EP 624 154, and particularly preferred in that family is acetyl triethyl citrate (ATC). Acetyl triethyl citrate has the advantage that it is environmentally friendly as it eventually degrades into citric acid and alcohol. Furthermore, acetyl triethyl citrate has a good hydrolytical stability in the product upon storage and it is an efficient bleach activator. Finally, it provides good building capacity to the composition.

The compositions according to the present invention comprise from 0.1% to 20% by weight of the total composition of said liquid hydrophobic bleach activator, or mixtures thereof, preferably from 1% to 10%, and more preferably from 1.5% to 7%.

As another essential ingredient, the compositions of the present invention comprise a long chain acyl sarcosinate or mixtures thereof, in its acid and/or salt form selected as desired for the compositions and uses herein, having the following formula:



wherein M is hydrogen or a cationic moiety and wherein R is an alkyl group of from 11 to 15 carbon atoms, preferably of from 11 to 13 carbon atoms. Preferred M are hydrogen and alkali metal salts, especially sodium and potassium. Said acyl sarcosinate surfactants are derived from natural fatty acids and the amino-acid sarcosine (N-methyl glycine). They are suitable to be used as aqueous solution of their salt or in their acidic form as powder. Being derivatives of natural fatty acids, said acyl sarcosinates are rapidly and completely biodegradable and have good skin compatibility.

Accordingly particularly preferred long chain acyl sarcosinates to be used herein include C12 acyl sarcosinate (i.e. an acyl sarcosinate according to the above formula wherein M is hydrogen and R is an alkyl group of 11 carbon atoms) and C14 acyl sarcosinate (i.e. an acyl sarcosinate according to the above formula wherein M is hydrogen and R is an alkyl group of 13 carbon atoms). C12 acyl sarcosinate is commercially available, for example, as Hamposyl L-30[®] supplied by Hampshire. C14 acyl sarcosinate is commercially available, for example, as Hamposyl M-30[®] supplied by Hampshire.

Typically, the compositions of the present invention comprise from 0.01% to 30% by weight of the total composition of said long chain acyl sarcosinate, or mixtures thereof, preferably from 0.1% to 20%, and more preferably from 0.5% to 15%.

It has now been found that by adding said long chain acyl sarcosinate in a liquid composition comprising a peroxygen bleach and a liquid hydrophobic bleach activator, improved stain removal performance is obtained with said com-

position when used to pretreat a soiled fabric before said fabric is washed, as compared to the stain removal performance delivered with the same composition without said long chain sarcosinate, or to the same composition with another anionic hydrophilic surfactant like for instance C12 alkyl sulfate, or C12-C15 alkyl-ethoxy sulphate (e.g., C25AE2.5S) instead of said long chain acyl sarcosinate.

By "stain removal performance" it is meant herein stain removal performance on a variety of stains/soils such as greasy/oily stains and/or bleachable stains and/or enzymatic stains.

By "greasy/oily stains" it is meant herein any soil and stain of greasy nature that can be found on a fabric like dirty motor oil, mineral oil, make-up, lipstick vegetal oil, spaghetti sauce, mayonnaise, chocolate and the like.

Examples of bleachable stains include tea, coffee, wine and the like. An example of enzymatic stains includes blood.

The stain removal performance of a composition on a soiled fabric under pretreatment conditions may be evaluated by the following test method. A composition according to the present invention is first applied to a fabric, preferably to the stained portion of said fabric, left to act from about 1 to about 10 minutes, preferably 5 minutes, and said pretreated fabric is then washed according to common washing conditions, at a temperature of from 30°C to 70° C for a period of time sufficient to bleach said fabric. The stain removal performance is then evaluated by comparing side by side the soiled fabric pretreated with the composition according to the present invention with those pretreated with the reference, e.g. the same compositions but without any long chain (C12-C16) acyl sarcosinates. A visual grading scale may be used to assign differences in panel score units (psu), in a range from 0 to 4.

Actually, it has now been found that improved stain removal performance, is achieved with said long chain (C12-C16) acyl sarcosinates when contacting them to fabrics under acidic to neutral pH conditions. This because the optimum lowering of surface tension by said acyl sarcosinate surfactants is achieved in the weakly acid to neutral pH range, i.e., in the pH conditions of the compositions according to the present invention. Thus the stain removal performance associated to said long chain acyl sarcosinates is not peroxygen bleach dependent. It has also been observed that by lowering or highering the chain length of said long chain acyl sarcosinates according to the present invention, the stain removal performance under pretreatment conditions according to the present invention decreases.

Thus, in its broadest aspect the present invention encompasses the use of a long chain (C12-C16) acyl sarcosinate, in a liquid composition having a pH up to 7, preferably in a peroxygen bleach-containing liquid composition, for improving the stain removal performance of said composition under pretreatment conditions, i.e., when pretreating a soiled fabric with said composition before said fabric is washed.

The peroxygen bleach-containing compositions according to the present invention comprise a liquid hydrophobic bleach activator and are thus formulated either as aqueous emulsions of said liquid hydrophobic bleach activator in a matrix comprising water, the peroxygen bleach and an emulsifying surfactant system comprising said long chain acyl sarcosinate (as a hydrophilic surfactant having an HLB above 10), or as microemulsions of said liquid hydrophobic bleach activator in a matrix comprising water, the peroxygen bleach and a hydrophilic surfactant system comprising at least said long chain acyl sarcosinate.

In the embodiment of the present invention where the peroxygen bleach-containing compositions of the present invention are formulated as aqueous emulsions, said long chain acyl sarcosinate may be used, as sole emulsifying surfactant, to emulsify said hydrophobic bleach activator. Accordingly, if said long chain acyl sarcosinate is present in said emulsions as the sole emulsifying surfactant, it is preferably present at a level of from 1% to 6% by weight of the total composition.

Preferred peroxygen bleach-containing emulsions herein comprise an emulsifying surfactant system of at least two different surfactants, i.e. at least a hydrophobic surfactant having an HLB up to 9.5 or mixtures thereof, and at least a hydrophilic surfactant having an HLB above 10 or mixtures thereof, in order to emulsify the liquid hydrophobic bleach activator. Indeed, said two different surfactants in order to form emulsions which are stable must have different HLB values (hydrophilic / lipophilic balance), and preferably the difference in value of the HLBs of said two surfactants is at least 1, preferably at least 2. In other words, by appropriately combining at least two of said surfactants with different HLBs in water, stable emulsions will be formed, i.e. emulsions which do not substantially separate into distinct layers, upon standing for at least two weeks at 40°C.

The preferred emulsions according to the present invention comprise from 1 % to 50 % by weight of the total composition of said hydrophilic and hydrophobic surfactants, more preferably from 5 % to 40 % and most preferably from 8 % to 30 %. The preferred emulsions according to the present invention comprise at least from 0.01 % by weight of the total emulsion of said hydrophobic surfactant, or mixtures thereof, preferably at least 2 % and more preferably at least 4 % and at least from 0.01 % by weight of the total emulsion of said hydrophilic surfactant, or mixtures thereof, preferably at least 2%, and more preferably at least 4%.

The hydrophilic surfactants having an HLB above 10 to be used in said emulsions herein are said long chain acyl sarcosinates alone or in mixture with other hydrophilic surfactants having an HLB above 10, preferably hydrophilic non-ionic surfactants having an HLB above 10 and more preferably above 10.5. Preferred to be used herein as the hydrophobic surfactants are the hydrophobic nonionic surfactants. Said hydrophobic nonionic surfactants to be used herein have an HLB up to 9.5, preferably below 9.5, more preferably below 9. Indeed, the hydrophobic nonionic surfactants to

be used herein have excellent grease cutting properties, i.e. they have a solvent effect which contributes to hydrophobic soils removal. The hydrophobic surfactants act as carrier of the hydrophobic brighteners onto the fabrics allowing thereby said brighteners to work in close proximity with the fabrics surface since the beginning of the wash.

Suitable nonionic surfactants for use herein include alkoxyated fatty alcohols preferably, fatty alcohol ethoxylates and/or propoxylates. Indeed, a great variety of such alkoxyated fatty alcohols are commercially available which have very different HLB values (hydrophilic / lipophilic balance). The HLB values of such alkoxyated nonionic surfactants depend essentially on the chain length of the fatty alcohol, the nature of the alkoxylation and the degree of alkoxylation. Hydrophilic nonionic surfactants tend to have a high degree of alkoxylation and a short chain fatty alcohol, while hydrophobic surfactants tend to have a low degree of alkoxylation and a long chain fatty alcohol. Surfactants catalogs are available which list a number of surfactants including nonionics, together with their respective HLB values.

Suitable chemical processes for preparing the nonionic surfactants for use herein include condensation of corresponding alcohols with alkylene oxide, in the desired proportions. Such processes are well known to the man skilled in the art and have been extensively described in the art. As an alternative, a great variety of alkoxyated alcohols suitable for use herein is commercially available from various suppliers.

Preferred hydrophobic nonionic surfactants to be used in the emulsions according to the present invention are surfactants having an HLB up to 9 and being according to the formula $RO-(C_2H_4O)_n(C_3H_6O)_mH$, wherein R is a C_6 to C_{22} alkyl chain or a C_6 to C_{28} alkyl benzene chain, and wherein $n+m$ is from 0.5 to 5 and n is from 0 to 5 and m is from 0 to 5 and preferably $n+m$ is from 0.5 to 4.5 and, n and m are from 0 to 4.5. The preferred R chains for use herein are the C_8 to C_{22} alkyl chains. Accordingly suitable hydrophobic nonionic surfactants for use herein are Dobanol^R 91-2.5 (HLB= 8.1; R is a mixture of C_9 and C_{11} alkyl chains, n is 2.5 and m is 0), or Lutensol^R TO3 (HLB=8; R is a mixture of C_{13} and C_{15} alkyl chains, n is 3 and m is 0), or Tergitol^R 25L3 (HLB= 7.7; R is in the range of C_{12} to C_{15} alkyl chain length, n is 3 and m is 0), or Dobanol^R 23-3 (HLB=8.1; R is a mixture of C_{12} and C_{13} alkyl chains, n is 3 and m is 0), or Dobanol^R 23-2 (HLB= 6.2; R is a mixture of C_{12} and C_{13} alkyl chains, n is 2 and m is 0), or mixtures thereof. Preferred herein are Dobanol^R 23-3, or Dobanol^R 23-2, Lutensol^R TO3, or mixtures thereof. These Dobanol^R surfactants are commercially available from SHELL. These Lutensol^R surfactants are commercially available from BASF and these Tergitol^R surfactants are commercially available from UNION CARBIDE. Other suitable hydrophobic nonionic surfactants to be used herein are non alkoxyated surfactants. An example is Dobanol^R 23 (HLB<3).

Preferred hydrophilic nonionic surfactants to be used in the emulsions according to the present invention are surfactants having an HLB above 10 and being according to the formula $RO-(C_2H_4O)_n(C_3H_6O)_mH$, wherein R is a C_6 to C_{22} alkyl chain or a C_6 to C_{28} alkyl benzene chain, and wherein $n+m$ is from 5 to 11 and n is from 0 to 11 and m is from 0 to 11, preferably $n+m$ is from 6 to 10 and, n and m are from 0 to 10. Throughout this description n and m refer to the average degree of the ethoxylation/propoxylation. The preferred R chains for use herein are the C_8 to C_{22} alkyl chains. Accordingly suitable hydrophilic nonionic surfactants for use herein are Dobanol^R 23-6.5 (HLB=11.9; R is a mixture of C_{12} and C_{13} alkyl chains, n is 6.5 and m is 0), or Dobanol^R 25-7 (HLB=12; R is a mixture of C_{12} to C_{15} alkyl chains, n is 7 and m is 0), or Dobanol^R 45-7 (HLB=11.6; R is a mixture of C_{14} and C_{15} alkyl chains, n is 7 and m is 0), or Dobanol^R 91-5 (HLB=11.6; R is a mixture of C_9 to C_{11} alkyl chains, n is 5 and m is 0), or Dobanol^R 91-6 (HLB=12.5; R is a mixture of C_9 to C_{11} alkyl chains, n is 6 and m is 0), or Dobanol^R 91-8 (HLB=13.7; R is a mixture of C_9 to C_{11} alkyl chains, n is 8 and m is 0), or Dobanol^R 91-10 (HLB= 14.2; R is a mixture of C_9 to C_{11} alkyl chains, n is 10 and m is 0), or mixtures thereof. Preferred herein are Dobanol^R 91-10, or Dobanol^R 45-7, Dobanol^R 23-6.5, or mixtures thereof. These Dobanol^R surfactants are commercially available from SHELL. Apart from the hydrophilic nonionic surfactants other hydrophilic surfactants may further be used in the emulsions of the present invention such as anionic surfactants described hereinafter.

The emulsions according to the present invention may further comprise other surfactants on top of said long chain acyl sarcosinate alone or in an emulsifying system further comprising hydrophobic surfactants and optional other hydrophilic surfactants, which should however not significantly alter the weighted average HLB value of the overall emulsion.

In a particularly preferred embodiment of the emulsions of the present invention, wherein the emulsions comprise acetyl triethyl citrate as the bleach activator, an adequate nonionic emulsifying surfactant system on top of said long chain acyl sarcosinate, would comprise a hydrophobic nonionic surfactant with for instance an HLB of 6, such as a Dobanol^R 23-2 and a hydrophilic nonionic surfactant with for instance an HLB of 15, such as a Dobanol^R 91-10. Other suitable nonionic surfactant systems comprise for example a Dobanol^R 23-6.5 (HLB about 12) and a Dobanol^R 23 (HLB below 6) or a Dobanol^R 45-7 (HLB=11.6) and a Dobanol 23-3 (HLB=8.1).

The preferred making of the preferred emulsions of the present invention, which comprise a liquid hydrophobic bleach activator, includes: (i) premixing the hydrophobic surfactants with the bleach activator and other non-water miscible ingredients if present; (ii) premixing the hydrophilic surfactants (i.e. said long chain acyl sarcosinates with optional others hydrophilic surfactants like hydrophilic nonionic surfactants) with water and subsequently adding the other water soluble ingredients if present; (iii) trimming the pH to preferred value; (iv) including a peroxygen bleach, e.g., hydrogen peroxide, and the hydrophobic phase prepared in step (i). It is important that during the mixing of the two phases, the emulsions be constantly kept under stirring under relatively low stirring energies, preferably 30 minutes at 550 rpm,

most preferably 30 minutes at 450 rpm.

In the embodiment of the present invention where the compositions are formulated as emulsions said compositions are opaques. In centrifugation examination, it was observed that said emulsions herein showed no phase separation after 15 minutes at 6000 rpm. Under microscopic examination, said emulsions appeared as a dispersion of droplets in a matrix.

In the embodiment of the present invention where the peroxygen bleach-containing compositions of the present invention are formulated as microemulsions, said peroxygen bleach-containing microemulsions according to the present invention comprise a hydrophilic surfactant system comprising said long chain acyl sarcosinate. Accordingly, if said acyl sarcosinate is present in said microemulsions as the sole emulsifying surfactant, it is preferably present at a level of more than 6% of the total composition up to 15%. A key factor in order to stably incorporate the hydrophobic bleach activator is that said long chain acyl sarcosinate has a different HLB value to that of the hydrophobic activator. It has also been observed that the long chain acyl sarcosinate herein, even at low levels, have shown to improve the physical stability of the microemulsions of the present invention, even at higher temperatures (up to 40°C).

The hydrophilic surfactant system emulsifying said hydrophobic bleach activator in the microemulsions of the present invention may further comprise other hydrophilic surfactants like nonionic hydrophilic surfactants and/or other anionic surfactants. A key factor in order to stably incorporate the hydrophobic activator in said microemulsions is that at least one of said surfactants of the hydrophilic surfactant system must have a different HLB value to that of the hydrophobic activator. Indeed, if all said surfactants had the same HLB value as that of the hydrophobic activator, a continuous single phase might be formed thus lowering the chemical stability of the bleach/bleach activator system. Preferably, at least one of said surfactants has an HLB value which differs by at least 1.0 HLB unit, preferably 2.0 to that of said bleach activator.

Suitable other anionic surfactants to be further added in the compositions herein include water soluble salts or acids of the formula ROSO_3M wherein R preferably is a $\text{C}_{10}\text{-C}_{24}$ hydrocarbyl, preferably an alkyl or hydroxyalkyl having a $\text{C}_{10}\text{-C}_{20}$ alkyl component, more preferably a $\text{C}_{12}\text{-C}_{18}$ alkyl or hydroxyalkyl, and M is H or a cation, e.g., an alkali metal cation (e.g., sodium, potassium, lithium), or ammonium or substituted ammonium (e.g., methyl-, dimethyl-, and trimethyl ammonium cations and quaternary ammonium cations, such as tetramethyl-ammonium and dimethyl piperdinium cations and quaternary ammonium cations derived from alkylamines such as ethylamine, diethylamine, triethylamine, and mixtures thereof, and the like). Typically, alkyl chains of $\text{C}_{12}\text{-C}_{16}$ are preferred for lower wash temperatures (e.g., below about 50°C) and $\text{C}_{16}\text{-C}_{18}$ alkyl chains are preferred for higher wash temperatures (e.g., above about 50°C).

Other suitable anionic surfactants for use herein are water soluble salts or acids of the formula $\text{RO(A)}_m\text{SO}_3\text{M}$ wherein R is an unsubstituted $\text{C}_{10}\text{-C}_{24}$ alkyl or hydroxyalkyl group having a $\text{C}_{10}\text{-C}_{24}$ alkyl component, preferably a $\text{C}_{12}\text{-C}_{20}$ alkyl or hydroxyalkyl, more preferably $\text{C}_{12}\text{-C}_{18}$ alkyl or hydroxyalkyl, A is an ethoxy or propoxy unit, m is greater than zero, typically between about 0.5 and about 6, more preferably between about 0.5 and about 3, and M is H or a cation which can be, for example, a metal cation (e.g., sodium, potassium, lithium, calcium, magnesium, etc.), ammonium or substituted-ammonium cation. Alkyl ethoxylated sulfates as well as alkyl propoxylated sulfates are contemplated herein. Specific examples of substituted ammonium cations include methyl-, dimethyl-, trimethyl-ammonium and quaternary ammonium cations, such as tetramethyl-ammonium, dimethyl piperdinium and cations derived from alkanolamines such as ethylamine, diethylamine, triethylamine, mixtures thereof, and the like. Exemplary surfactants are $\text{C}_{12}\text{-C}_{18}$ alkyl polyethoxylate (1.0) sulfate, $\text{C}_{12}\text{-C}_{18}\text{E(1.0)M}$, $\text{C}_{12}\text{-C}_{18}$ alkyl polyethoxylate (2.25) sulfate, $\text{C}_{12}\text{-C}_{18}\text{E(2.25)M}$, $\text{C}_{12}\text{-C}_{18}$ alkyl polyethoxylate (3.0) sulfate $\text{C}_{12}\text{-C}_{18}\text{E(3.0)}$, and $\text{C}_{12}\text{-C}_{18}$ alkyl polyethoxylate (4.0) sulfate $\text{C}_{12}\text{-C}_{18}\text{E(4.0)M}$, wherein M is conveniently selected from sodium and potassium.

Other anionic surfactants useful for deterative purposes can also be used herein. These can include salts (including, for example, sodium, potassium, ammonium, and substituted ammonium salts such as mono-, di- and triethanolamine salts) of soap, $\text{C}_9\text{-C}_{20}$ linear alkylbenzenesulfonates, $\text{C}_8\text{-C}_{22}$ primary or secondary alkanesulfonates, $\text{C}_8\text{-C}_{24}$ olefinsulfonates, sulfonated polycarboxylic acids prepared by sulfonation of the pyrolyzed product of alkaline earth metal citrates, e.g., as described in British patent specification No. 1,082,179, $\text{C}_8\text{-C}_{24}$ alkylpolyglycol ethersulfates (containing up to 10 moles of ethylene oxide); alkyl ester sulfonates such as $\text{C}_{14}\text{-C}_{16}$ methyl ester sulfonates; acyl glycerol sulfonates, fatty oleyl glycerol sulfates, alkyl phenol ethylene oxide ether sulfates, paraffin sulfonates, alkyl phosphates, isethionates such as the acyl isethionates, N-acyl taurates, alkyl succinamates and sulfosuccinates, monoesters of sulfosuccinate (especially saturated and unsaturated $\text{C}_{12}\text{-C}_{18}$ monoesters) diesters of sulfosuccinate (especially saturated and unsaturated $\text{C}_6\text{-C}_{14}$ diesters), sulfates of alkylpolysaccharides such as the sulfates of alkylpolyglucoside (the nonionic nonsulfated compounds being described below), branched primary alkyl sulfates, alkyl polyethoxy carboxylates such as those of the formula $\text{RO}(\text{CH}_2\text{CH}_2\text{O})_k\text{CH}_2\text{COO-M}^+$ wherein R is a $\text{C}_8\text{-C}_{22}$ alkyl, k is an integer from 0 to 10, and M is a soluble salt-forming cation. 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 tall oil. Further examples are given in "Surface Active Agents and Detergents" (Vol. I and II by Schwartz, Perry and Berch). A variety of such surfactants are also generally disclosed in U.S. Patent 3,929,678, issued December 30, 1975 to Laughlin, et al. at Column 23, line 58 through Column 29, line 23 (herein incorporated by reference).

Suitable nonionic surfactants for use in the microemulsions herein include the hydrophilic nonionic surfactants as

defined herein before for the emulsions.

The preferred making of the microemulsions of the present invention which comprises a liquid hydrophobic bleach activator includes premixing the surfactants with water and subsequently adding the other ingredients including the per-
 5 oxygen bleach, e.g., hydrogen peroxide, and said hydrophobic bleach activator. Irrespective of this preferred order of addition, it is important that during the mixing of the ingredients, the microemulsions be constantly kept under stirring under relatively high stirring energies, preferably 30 minutes at 750 rpm, most preferably 30 minutes at 1000 rpm.

In the embodiment of the present invention where the compositions are formulated as microemulsions said compositions are macroscopically transparent in the absence of opacifiers and dyes. In centrifugation examination, it was
 10 observed that said microemulsions herein showed no phase separation after 15 minutes at 6000 rpm. Under microscopic examination, said microemulsions appeared as a dispersion of droplets in a matrix. The matrix is the hydrophilic matrix described hereinbefore, and the droplets are constituted by the liquid hydrophobic bleach activator. We have observed that the particles had a size which is typically around or below 3 micron diameter.

The peroxygen bleach-containing compositions of the present invention formulated in the form of emulsions or microemulsions are chemically stable. By "chemically stable" it is meant herein that said composition of the present
 15 invention comprising a peroxygen bleach does not undergo more than 10% available oxygen loss at 50°C in 2 weeks. The concentration of available oxygen can be measured by chemical titration methods known in the art, such as the iodimetric method, the permanganometric method and the cerimetric method. Said methods and the criteria for the choice of the appropriate method are described for example in "Hydrogen Peroxide", W. C. Schumb, C. N. Satterfield and R. L. Wentworth, Reinhold Publishing Corporation, New York, 1955 and "Organic Peroxides", Daniel Swern, Editor
 20 Wiley Int. Science, 1970. Alternatively the stability of said compositions may also be evaluated by a bulging test method.

Accordingly said peroxygen bleach-containing compositions of the present invention may be packaged in a given deformable container/bottle without compromising the stability of said container/bottle comprising it upon standing, for long periods of time.

The peroxygen-bleach containing compositions of the present invention formulated either as an emulsion or as a
 25 microemulsion may further comprise other surfactants known to those skilled in the art including cationic, zwitterionic and/or amphoteric surfactants. For example, the peroxygen bleach-containing compositions of the present invention may further comprise an amine oxide surfactant according to the formula $R_1R_2R_3NO$, wherein each of R_1 , R_2 and R_3 is independently a C_1 - C_{30} , preferably a C_1 - C_{20} , most preferably a C_1 - C_{16} hydrocarbon chain. Indeed, we have observed that improved chemical stability, i.e., lower decomposition of the bleach and the bleach activator is obtained by adding
 30 such an amine oxide. It is believed that such stability is due to the capacity of the amine-oxide to limit interactions between the bleach and the bleach activator possibly through emulsification. It is believed that this stabilising effect is matrix independent. Amine oxides may be preferably present in amounts up to 10 % by weight of the total composition, more preferably from 1% to 3%.

The compositions according to the present invention are aqueous liquid cleaning compositions. Said aqueous compositions should be formulated in the acidic pH up to the neutral pH ($pH=7$), preferably at a pH of from 0 to 6 and more preferably at a pH of from 1 to 5. Formulating the compositions of the present invention in the acidic pH range contributes to the stability of said compositions. The pH of the compositions of the present invention can be adjusted by using organic or inorganic acids, or alkalising agents.

The compositions of the present invention may further comprise optional ingredients like stabilisers, cheating
 40 agents, radical scavengers, builders, soil suspenders, dye transfer agents, solvents, brighteners, perfumes, foam suppressors or dyes or mixtures thereof.

The peroxygen bleach-containing compositions of the present invention may comprise a chelating agent or mixtures thereof, as a highly preferred optional ingredient. Chelating agents suitable to be used herein include chelating agents selected from the group of phosphonate chelating agents, amino carboxylate cheating agents, polyfunctionally-
 45 substituted aromatic chelating agents, and further chelating agents like glycine, salicylic acid, aspartic acid, glutamic acid, malonic acid, or mixtures thereof. Chelating agents when used, are typically present herein in amounts ranging from 0.001% to 5% by weight of the total composition and preferably from 0.05% to 2% by weight.

Suitable phosphonate chelating agents to be used herein may include ethydrionic acid as well as amino phosphonate compounds, including amino alkylene poly (alkylene phosphonate), alkali metal ethane 1-hydroxy diphosphonates, nitrilo trimethylene phosphonates, ethylene diamine tetra methylene phosphonates, and diethylene triamine penta methylene phosphonates. The phosphonate compounds may be present either in their acid form or as salts of different cations on some or all of their acid functionalities. Preferred phosphonate cheating agents to be used herein are diethylene triamine penta methylene phosphonates. Such phosphonate chelating agents are commercially available from Monsanto under the trade name DEQUEST[®].

Polyfunctionally-substituted aromatic chelating agents may also be useful in the compositions herein. See U.S. patent 3,812,044, issued May 21, 1974, to Connor et al. Preferred compounds of this type in acid form are dihydroxydisulfobenzenes such as 1,2-dihydroxy -3,5-disulfobenzene.

A preferred biodegradable chelating agent for use herein is ethylene diamine N,N'- disuccinic acid, or alkali metal, or alkaline earth, ammonium or substitutes ammonium salts thereof or mixtures thereof. Ethylenediamine N,N'- disuc-

cinic acids, especially the (S,S) isomer have been extensively described in US patent 4, 704, 233, November 3, 1987. to Hartman and Perkins. Ethylenediamine N,N'-disuccinic acids is, for instance, commercially available under the trade-name ssEDDS[®] from Palmer Research Laboratories.

Suitable amino carboxylates to be used herein include ethylene diamine tetra acetates, diethylene triamine pentaacetates, diethylene triamine pentaacetate (DTPA), N-hydroxyethylethylenediamine triacetates, nitrilotri-acetates, ethylenediamine tetrapropionates, triethylenetetraaminehexa-acetates, ethanoldiglycines, propylene diamine tetracetic acid (PDTA) and methyl glycine di-acetic acid (MGDA), both in their acid form, or in their alkali metal, ammonium, and substituted ammonium salt forms. Particularly suitable amino carboxylates to be used herein are diethylene triamine pentaacetic acid, propylene diamine tetracetic acid (PDTA) which is, for instance, commercially available from BASF under the trade name Trilon FS[®] and methyl glycine diacetic acid (MGDA).

Particularly preferred chelating agents to be used herein are diethylene triamine methylene phosphonate, ethylene N,N'-disuccinic acid, diethylene triamine pentaacetate, glycine, salicylic acid, aspartic acid, glutamic acid, malonic acid or mixtures thereof and highly preferred is salicylic acid. Salicylic acid may be commercially available from Rhone-Poulenc under the name Salicylic Acid[®].

The peroxygen bleach-containing compositions of the present invention may further comprise a radical scavenger, or mixtures thereof, as a highly preferred optional ingredient. Suitable radical scavengers for use herein include the well-known substituted mono and di hydroxy benzenes and their analogs, alkyl and aryl carboxylates and mixtures thereof. Preferred such radical scavengers for use herein include di-tert-butyl hydroxy toluene (BHT), hydroquinone, di-tert-butyl hydroquinone, mono-tert-butyl hydroquinone, tert-butyl-hydroxy anisole, benzoic acid, toluic acid, catechol, t-butyl catechol, benzylamine, 1,1,3-tris(2-methyl-4-hydroxy-5-t-butylphenyl) butane, n-propyl-gallate or mixtures thereof and highly preferred is di-tert-butyl hydroxy toluene. Radical scavengers when used, are typically present herein in amounts ranging from 0.001% to 2% by weight of the total composition and preferably from 0.001% to 0.5% by weight.

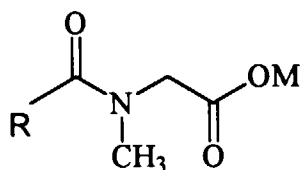
The presence of chelating agents and/or radical scavengers allows to provide compositions that are particularly safe to the fabrics treated therewith and to the color, even when used for pretreating a soiled fabric upon prolonged contact times before washing said fabric.

The peroxygen bleach-containing compositions of the present invention may further comprise up to 10%, preferably from 2% to 4% by weight of the total composition of an alcohol according to the formula HO - CR'R'' - OH, wherein R' and R'' are independently H or a C₂-C₁₀ hydrocarbon chain and/or cycle. Preferred alcohol according to that formula is propanediol. Indeed, we have observed that these alcohols in general and propanediol in particular also improve the chemical stability of the compositions, i.e. lower the decomposition of the bleach and the bleach activator, as the amine oxides herein above. In addition, said alcohols lower the surface tension of the product, thus preventing superficial film or gel formation. Thus said alcohols improve the aesthetics of the compositions herein. It is believed that the chemical stabilising effect of said alcohols is twofold. Firstly, they may work as radical scavengers and secondly, they may interact with the hydrogen peroxide preventing or limiting hydrolysis, therefore reducing the rate of peroxide decomposition. It is believed that this improvement in chemical stability obtained by said alcohols is matrix independent.

The peroxygen bleach-containing compositions according to the present invention may further comprise a foam suppressors such as 2-alkyl alkanol, or mixtures thereof, as an optional ingredient. Particularly suitable to be used in the present invention are the 2-alkyl alkanols having an alkyl chain comprising from 6 to 16 carbon atoms, preferably from 8 to 12 and a terminal hydroxy group, said alkyl chain being substituted in the α position by an alkyl chain comprising from 1 to 10 carbon atoms, preferably from 2 to 8 and more preferably 3 to 6. Such suitable compounds are commercially available, for instance, in the Isofol[®] series such as Isofol[®] 12 (2-butyl octanol) or Isofol[®] 16 (2-hexyl decanol). Typically, the compositions of the present invention comprise up to 2 % by weight of the total composition of a 2-alkyl alkanol, or mixtures thereof, preferably from 0.05 % to 1.5 % and more preferably from 0.1 % to 0.8 %.

Although preferred application of the compositions described herein is laundry pretreatment, the compositions according to the present invention may also be used as a laundry detergent or as a laundry detergent booster and as a household cleaner in the bathroom or in the kitchen.

The present invention further encompasses a process of pretreating soiled fabrics with a liquid composition having a pH up to 7 and comprising a long chain acyl sarcosinate having the following formula



wherein M is hydrogen or a cationic moiety and wherein R is an alkyl group of 11 to 15 carbon atoms, said process comprises the steps of applying said composition in its neat form onto the fabric and allowing said composition to remain in

contact with said fabric preferably without leaving said composition to dry onto said fabric, before said fabric is washed. Said composition may remain in contact with said fabric, typically for a period of 1 minute to several hours, preferably 1 minute to 1 hour, more preferably 1 minutes to 30 minutes, and most preferably 2 to 10 minutes. Optionally, when the fabric is soiled with encrusted stains/soils which otherwise would be relatively difficult to remove, said compositions may be rubbed and/or brushed more or less intensively, for example, by means of a sponge or a brush or simply by rubbing two pieces of fabric each against the other. In the preferred process of pretreating soiled fabrics herein the compositions according to the present invention are used.

By "washing" it is to be understood herein to simply rinse the fabrics with water, or the fabrics may be washed with conventional compositions comprising at least one surface active agent, this by the means of a washing machine or simply by hand.

By "in its neat form" it is to be understood that the liquid compositions are applied directly onto the fabrics to be pretreated without undergoing any dilution, e.g., the compositions according to the present invention are applied as described herein.

According to the process of pretreating soiled fabrics of the present invention, the liquid compositions according to the present invention used in said process should preferably not be left to dry onto the fabrics. Indeed, it has been found that water evaporation contributes to increase the concentration of free radicals onto the surface of the fabrics and, consequently, the rate of chain reaction. It is also speculated that an auto-oxidation reaction occurs upon evaporation of water when the liquid compositions are left to dry onto the fabrics. Said reaction of auto-oxidation generates peroxy-radicals which may contribute to the degradation of cellulose. Thus, not leaving said liquid peroxygen bleach-containing compositions to dry onto the fabric, in the process of pretreating soiled fabrics according to the present invention, may reduce fabric damage what may otherwise result from prolonged contact time between said fabrics and liquid peroxygen bleach-containing compositions.

The present invention will be further illustrated by the following examples.

Examples

The following compositions were made by mixing the listed ingredients in the listed proportions (weight % unless otherwise specified).

Compositions	I	II	III	IV	V	VI	VII	VIII	IX	X	XI
Hamposyl® L-30	3.0	-	4.0	-	2.0	-	-	-	12.0	-	-
Hamposyl® M-30	-	3.0	-	4.0	2.0	3.0	3.0	12.0	-	12.0	6.0
Dobanol® 45-7	-	-	6.4	6.4	6.4	5.0	5.0	-	6.0	6.0	6.0
Dobanol® 23-3	-	-	8.6	8.6	8.6	8.6	7.3	-	-	-	-
Dobanol® 23-6.5	-	-	-	-	-	-	-	-	6.0	6.0	6.0
C25-AE-2.5-S	-	-	-	-	-	1.0	2.0	-	-	-	6.0
ATC*	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5
H ₂ O ₂	4.0	4.0	4.0	4.0	4.0	4.0	4.0	6.0	6.0	6.0	6.0
Water and minors	-----up to 100%-----										
H ₂ SO ₄ up to pH 4											

* ATC is acetyl triethyl citrate.

Dobanol ® 23-3 is a C12-C13 nonionic ethoxylated surfactant with HLB of 8.1.

Dobanol ® 23-6.5 is a C12-C13 nonionic ethoxylated surfactant with HLB of 11.9.

Dobanol ® 45-7 is a C14-C15 nonionic ethoxylated surfactant with HLB of 11.6.

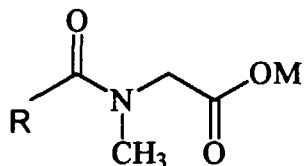
Hamposyl L-30® is C12 acyl sarcosinate commercially available from Hampshire.

Hamposyl M-30® is C14 acyl sarcosinate commercially available from Hampshire.

Compositions I to VII are emulsions according to the present invention and compositions VIII to XI are microemulsions according to the present invention. Excellent stain removal performance has been obtained on a variety of stains including greasy/oily stains like dirty motor oil, make-up, lipstick, spaghetti sauce, mayonnaise, bleachable stains like coffee, grass, and enzymatic stains like blood, when pretreating soiled fabrics with said compositions, e.g., when leaving said compositions to act onto the fabrics for a contact period of about 5 minutes, before washing said fabrics.

Claims

1. A liquid composition comprising a peroxygen bleach, or a source thereof, a liquid hydrophobic bleach activator, a long chain acyl sarcosinate having the following formula

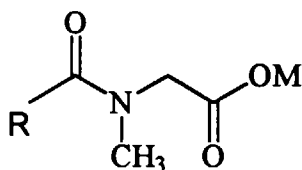


wherein M is hydrogen or a cationic moiety and wherein R is an alkyl group of from 11 to 15 carbon atoms, or mixtures thereof, said composition being formulated either as an emulsion or as a microemulsion.

2. A composition according to claim 1 wherein said long chain acyl sarcosinate is C12 acyl sarcosinate and/or C14

acyl sarcosinate.

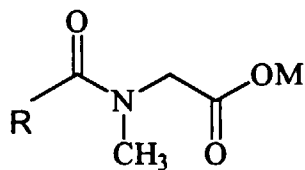
3. A composition according to any of the preceding claims wherein said composition comprises from 0.01% to 30% by weight of the total composition of said long chain acyl sarcosinate, or mixtures thereof, preferably from 0.1% to 20%, and more preferably from 0.5% to 15%.
4. A composition according to any of the preceding claims wherein said liquid hydrophobic bleach activator is acetyl triethyl citrate.
5. A composition according to any of the preceding claims wherein said composition comprises from 0.1% to 20% by weight of the total composition of said liquid hydrophobic bleach activator, or mixtures thereof, preferably from 1% to 10% by weight, and more preferably from 1.5% to 7%.
6. A composition according to any of the preceding claims wherein said peroxygen bleach is hydrogen peroxide or a water soluble source thereof, or mixtures thereof.
7. A composition according to any of the preceding claims wherein said composition comprises from 0.01% to 20% by weight of the total composition of said peroxygen bleach or source thereof, or mixtures thereof, preferably from 1% to 15%, and more preferably from 2% to 10%.
8. A composition according to any of the preceding claims wherein said composition is formulated as a microemulsion of said hydrophobic liquid bleach activator in a matrix comprising water, said peroxygen bleach and a hydrophilic surfactant system comprising said long chain acyl sarcosinate.
9. A composition according to claim 8 wherein said hydrophilic surfactant system further comprises a nonionic surfactant and/or another anionic surfactant.
10. A composition according to claims 8 or 9, wherein at least one of said surfactants of said hydrophilic surfactant system have an HLB value that differs to that of said bleach activator, preferably by 1 HLB unit.
11. A composition according to any of the claims 1 to 7, wherein said composition is formulated as an aqueous emulsion of said hydrophobic liquid bleach activator in a matrix comprising water, said peroxygen bleach and an emulsifying surfactant system comprising said long chain acyl sarcosinate as a hydrophilic surfactant having an HLB above 10.
12. A composition according to claim 11, wherein the emulsifying system comprises at least a hydrophilic surfactant having an HLB above 10 or mixtures thereof, and at least a hydrophobic surfactant having an HLB up to 9.5 or mixtures thereof, preferably a hydrophobic nonionic surfactant having an HLB below 9.5, and most preferably below 9, wherein said hydrophobic bleach activator is emulsified by said emulsifying surfactants.
13. A composition according to any of the preceding claims, which has a pH of from 0 to 7, preferably of from 0 to 6, and more preferably of from 1 to 5.
14. A composition according to any of the preceding claims wherein said composition further comprises a chelating agent and/or a radical scavenger.
15. A process of pretreating a soiled fabric with a liquid composition having a pH up to 7 and comprising a long chain acyl sarcosinate having the following formula



wherein M is hydrogen or a cationic moiety and wherein R is an alkyl group of 11 to 15 carbon atoms, said process comprising the steps of applying said composition in its neat form onto said fabric, and allowing said composition

to remain in contact with said fabric before said fabric is washed.

16. The use of a long chain acyl sarcosinate having the following formula



wherein M is hydrogen or a cationic moiety and wherein R is an alkyl group of 11 to 15 carbon atoms, in a liquid composition having a pH up to 7, preferably in a liquid peroxygen bleach-containing composition, for improving the stain removal performance of said composition when pretreating a soiled fabric with said composition, before said fabric is washed.



European Patent
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EUROPEAN SEARCH REPORT

Application Number
EP 96 87 0012

DOCUMENTS CONSIDERED TO BE RELEVANT							
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)				
X	WO-A-95 33043 (PROCTER & GAMBLE) 7 December 1995 * claims 1,2; example 2 *	1,3,6,7	C11D3/39 C11D17/00 C11D1/10				
A	EP-A-0 109 279 (SURGIKOS INC) 23 May 1984 * claims *	1-3,6					
A	WO-A-93 12067 (PROCTER & GAMBLE) 24 June 1993 * claim 1; examples 6-10 *	1,4,5					
A	US-A-4 421 668 (COX ROGER B ET AL) 20 December 1983 * example 2 *	1-3					
A	WO-A-94 11483 (PROCTER & GAMBLE) 26 May 1994 * page 4, line 15 - page 5, line 34; claim 1 *	1,4,5					
A	EP-A-0 667 392 (JEYES GROUP PLC) 16 August 1995 * page 15, line 17 - line 25; tables 5,6 *	1-3	<table border="1"> <thead> <tr> <th colspan="2">TECHNICAL FIELDS SEARCHED (Int.Cl.6)</th> </tr> </thead> <tbody> <tr> <td>C11D</td> <td></td> </tr> </tbody> </table>	TECHNICAL FIELDS SEARCHED (Int.Cl.6)		C11D	
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