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#### (54)**Process of bleaching fabrics**

- The present invention relates to the bleaching of fabrics with a liquid acidic aqueous persulfate-containing composition. More particularly, the present invention relates to a process of bleaching fabrics which comprises the steps of:
- first contacting said fabrics with an alkaline solution having a pH above 7,
- optionally rinsing with water
- then contacting said fabrics with an acidic liquid aqueous composition having a pH below 7 and comprising a persulfate salt, in its neat or diluted form,
- and subsequently rinsing with water. Excellent bleaching and overall stain removal performance are delivered to the soiled fabrics so bleached while being safe to both the fabrics perse and colors.

#### Description

#### Technical Field

5 **[0001]** The present invention relates to the bleaching of fabrics with liquid aqueous acidic persulfate-containing bleaching compositions having a pH below 7.

#### Background of the invention

[0002] Liquid aqueous bleaching compositions suitable for laundry applications are well known in the art. It is also known that bleaching compositions relying on hypochlorite as the bleaching agent can be preferred over oxygen bleaches like hydrogen peroxide, mainly for performance reasons, especially at lower temperatures.

[0003] However, there are some limitations to the convenience of hypochlorite bleaches. In particular, it is well known from consumers that hypochlorite bleaching may cause yellowing and/or damage of the fabrics which are being bleached. This holds particularly true for synthetic fabrics and indeed there is a standing prejudice against using hypochlorite bleaches on synthetic fibers, as evidenced by warnings on labels of commercially available hypochlorite bleaches. Also, a variety of fabrics made of or containing synthetic fibers are labeled by their manufacturers as non-bleachable. Also another drawback associated to hypochlorite based bleaching compositions is that undesirable typical chlorine odour may be perceived by the consumer during and after use, e.g., on the hands of the user or fabrics that have been bleached with such compositions.

[0004] Color and fabric damage can be minimised by using milder oxygen bleaches such as persulfate salts. Advantageously, such persulfate salts also are odour free. Persulfate-containing compositions have been described in the art for the bleaching of fabrics (see for instance in US 3822 114, WO97/24429 or WO97/24430). Such compositions can be used directly in water or as an additive to a fully formulated cleaning composition to bleach fabrics. However, consumers are still not fully satisfactory when bleaching fabrics with such compositions and using them either alone or as a detergent additive, and are looking for improved overall stain removal performance on a variety of stains including greasy stains and enzymatic stains as well as further improved bleaching performance (e.g., whiteness) when bleaching fabrics.

**[0005]** It is therefore an object of the present invention to address the issues mentioned above, namely those of providing improved overall stain removal performance on a wide range of stains as well as improved bleaching performance when bleaching fabrics.

[0006] We have found that these issues are efficiently addressed when a particular process of bleaching fabrics with an acidic liquid aqueous composition (pH below 7) containing a persulfate salt is used. Indeed, it has been found that when in a process of bleaching fabrics with such a liquid persulfate containing composition the step of contacting the fabrics with this composition in its neat or diluted form, is first preceded by a step where the fabrics are contacted with an alkaline solution having a pH above 7, before being subsequently rinsed with water, improved bleaching performance as well as improved overall stain removal performance are delivered to the fabrics. Indeed, it has surprisingly been found that it is by performing this process of bleaching fabrics that both improved stain removal performance on a wide range of stains including greasy and enzymatic stains and improved bleaching performance are provided, as compared to the performance delivered with a conventional use of a liquid persulfate-containing composition as a laundry additive or as the sole laundry detergent.

**[0007]** Furthermore, it has been found that these improved bleaching performance and improved overall stain removal performance are provided with the processes according to the present invention even for acidic liquid aqueous persulfate-containing compositions having undergone long storage time before their use according to the processes herein.

[0008] In a preferred aspect of the process of bleaching fabrics according to the present invention the acidic liquid aqueous compositions used preferably comprise on top of the persulfate salt, a bleach activator being an aldehyde and/or ketone and/or halide activator compound for further improved bleaching performance and improved stain removal performance.

[0009] Advantageously, the laundry bleaching operations according to the present invention with such an acidic liquid aqueous persulfate-containing composition are safe to the fabrics perse and/or fabrics colour. Indeed, it has surprisingly been found that formulating the persulfate salt in an acidic liquid aqueous composition contributes to improved safety to both the fabrics and colour bleached therewith as compared to solid containing persulfate salt composition.

#### Summary of the invention

[0010] The present invention is a process of bleaching a fabric which comprises the steps of:

- first contacting said fabric with an alkaline solution having a pH above 7,

- then contacting said fabric with a liquid aqueous composition having a pH below 7 and comprising a persulfate salt, in its neat or diluted form,
- and subsequently rinsing with water.

#### 5 <u>Detailed Description</u>

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[0011] The present invention encompasses a process of bleaching fabrics as described herein after.

**[0012]** By "fabrics", it is to be understood any types of fabrics including for example clothes, curtains, drapes, bed linens, bath linens, table cloths, sleeping bags, tents, upholstered furniture and the like.

**[0013]** The process of bleaching fabrics herein is suitable for both natural fabrics and synthetic fabrics. By "natural" fabrics, it is meant fabrics made of cotton, viscose, linene, silk and/or wood. By "synthetic" fabrics, it is meant those made of synthetic fibers like polymeric fibers (polyamide, polyester, lycra<sup>®</sup> and elasthane<sup>®</sup>), and those made of both natural and synthetic fibers.

[0014] The process of bleaching fabrics according to the present invention comprises as a first essential step, the step of contacting the fabrics to be bleached with an alkaline solution having a pH above 7, preferably from 8 to 13, more preferably from 8.5 to 12 and most preferably from 9 to 11.5, before contacting the fabrics with an acidic liquid aqueous persulfate-containing composition (pH below 7) as described herein after and subsequently rinsing the fabrics with water.

[0015] By "alkaline solution", it is meant herein any aqueous or non-aqueous solution having the required pH. Typically, the alkaline solution used in the processes of bleaching fabrics according to the present invention is a liquid detergent composition that has the required alkaline pH in its neat form and/or in its diluted form, e.g., when diluted typically with water for example in a conventional hand washing operation or a conventional laundry washing machine operation, prior being contacted with the fabrics. In another aspect of the present invention, the alkaline solution may also be obtained with a granular detergent composition that is diluted typically with water (for example as mentioned above) prior being contacted to the fabrics.

[0016] As used herein "liquid" includes "pasty" compositions. The liquid detergent compositions for use herein may be aqueous or non-aqueous and are preferably aqueous compositions. Granular detergent compositions can be in "compact" form, the liquid detergent compositions can also be in a "concentrated" form. The "compact" form of the compositions used herein is best reflected by density and, in terms of composition, by the amount of inorganic filler salt. Inorganic filler salts are conventional ingredients of detergent compositions in granular or powder form. In conventional detergent compositions the filler salts are present in substantial amounts, typically 17% to 35% by weight of the total composition. In the "compact" compositions, the filler salt is present in amounts not exceeding 15% of the total composition, preferably not exceeding 10%, most preferably not exceeding 5% by weight of the composition. The inorganic filler salts such as meant in the granular detergent compositions for use herein are selected from alkali and alkaline earth metal salts of sulphates and chlorides. A preferred filler salt is sodium sulphate.

**[0017]** Typically, the density of the granular detergent compositions suitable for use in the first step of the processes herein ranges from 550 to 1000 g/litre, preferably from 600 to 950 g/litre of the composition measured at 20°C.

[0018] Typically, the liquid detergent compositions and/or granular detergent compositions suitable for use in the processes of bleaching fabrics according to the present invention comprise at least a surfactant or a mixture thereof. Any surfactant know to those skilled in the art may be suitable for such detergent compositions including anionic, nonionic, amphoteric, cationic and/or zwitterionic surfactants. Typically, the detergent compositions suitable for use in the processes of bleaching according to the present invention comprise from 0.1% to 60% by weight of the total composition of a surfactant or mixture thereof, preferably from 1% to 40%.

[0019] Such liquid detergent and/or granular detergent compositions may also comprise at least one or more other conventional detergent ingredients, preferably selected from builders (up to 80%), organic polymeric compounds (up to 20%), bleaching agents (up to 25%), bleach activators (up to 15%), enzymes (typically 0.0001% to 2%), suds suppressers (up to 4%), dispersants (up to 15%), lime-soap dispersants (up to 15%), soil suspending agents (up to 20%), antiredepositions agents (up to 20%), corrosion inhibitors (up to 10%), solvents (up to 20%), chelating agents (up to 15%), dyes, pigments, perfumes and the like. The precise nature of the additional conventional detergent ingredients and levels of incorporation thereof will depend on the physical form of the detergent composition and the end characteristics of the composition desired.

**[0020]** Suitable conventional detergent ingredients for use in such detergent compositions are described for example in co-pending patent applications UK 96/16112.0, UK 97/13946.3, or WO 96/28531 and/or EP-B-707626.

**[0021]** Typically, such liquid detergent compositions and/or granular detergent compositions may be diluted up to 250 times their weight of water to provide an alkaline solution as described herein, preferably from 2 to 200 and more preferably from 10 to 160. Such dilution may occur for instance in hand laundry application as well as by other means such as in washing machine.

[0022] Typically, in the first step of the process of bleaching fabrics according to the present invention the alkaline solu-

tion remains in contact with the fabrics typically up to 24 hours, preferably 5 minutes to 12 hours and more preferably from 20 minutes to 6 hours.

[0023] The process of bleaching fabrics according to the present invention comprises as a second essential step, the step of contacting the fabrics with an acidic liquid aqueous composition (pH below 7) comprising a persulfate salt or a mixture thereof.

[0024] The persulfate-containing compositions for use in the process of bleaching fabrics according to the present invention are liquid compositions as opposed to a solid or a gas. As used herein "liquid" includes "pasty" compositions. These liquid compositions herein are aqueous compositions. These liquid compositions according to the present invention are acidic, i.e. they have a pH below 7, preferably from 0.1 to 6, more preferably from 0.5 to 4, even more preferably from 1 to 3 and most preferably from 1 to 2. Formulating these compositions in the acidic pH range is critical to the chemical stability of these compositions upon prolonged periods of storage before their use in the process of bleaching fabrics according to the present invention.

[0025] The pH of these compositions may be adjusted by any acidifying or alkalinising agents known to those skilled in the art. Examples of acidifying agents are organic acids such as citric acid and inorganic acids such as sulphuric acid, sulphonic acid and/or methanesulphonic acid. Examples of alkalinising agents are sodium hydroxide, potassium hydroxide and/or sodium carbonate.

**[0026]** As an essential element, the acidic liquid aqueous compositions for use in the process of bleaching fabrics according to the present invention comprise a persulfate salt or a mixture thereof.

[0027] Suitable persulfate salts include any alkali metal persulfate salt including sodium persulfate salts and/or potassium persulfate salts. Preferred persulfate salt for use herein is the monopersulfate salt. Examples of monopersulfate salts commercially available are those commercialised by Interox under the trade name Curox<sup>®</sup>, by Degussa under the trade name Caroat<sup>®</sup> or by DuPont under the trade name Oxone<sup>®</sup>. It is to understand herein that when the commercially available Curox<sup>®</sup>, Caroat<sup>®</sup> and/or Oxone<sup>®</sup> are used, the % weights of persulfate salts mentioned herein, refer to the total weight of said Curox<sup>®</sup>, Caroat<sup>®</sup> and/or Oxone<sup>®</sup>. The active concentration is approximately 1/2 of the total weight. Other persulfate salts such as dipersulfate salts commercially available among others from Peroxide Chemie GMBH can be used in the compositions according to the present invention. Another suitable persulphate salt is ammonium persulphate.

**[0028]** Typically, the acidic liquid aqueous compositions for use in the process of bleaching fabrics according to the present invention comprise from 0.01% to 50% by weight of the total composition of the persulfate salt or mixtures thereof, preferably from 0.5% to 20%, more preferably from 1% to 15% and most preferably from 2% to 10%.

**[0029]** The process of bleaching fabrics according to the present invention comprises after the step of contacting the fabrics with an alkaline solution having a pH above 7 (the so called "alkaline step"), the step of contacting the fabrics with an acidic liquid aqueous composition comprising persulfate or a mixture thereof, in its neat form or diluted form (the so called "bleaching step").

[0030] By "diluted form", it is meant herein that the persulfate-containing composition as described herein after, may be diluted by the user, preferably with water. Such dilution may occur for instance in hand laundry applications as well as by the other means such as in washing machine. Said compositions can be diluted up to 500 times, preferably from 5 to 200 times and more preferably from 10 to 80 times. In a preferred embodiment herein said persulfate-containing composition is used in its diluted form.

[0031] In the process of bleaching fabrics according to the present invention the acidic liquid aqueous persulfate-containing composition remains in contact with the fabrics for a period of time sufficient to bleach the fabrics. When used in diluted form the acidic liquid aqueous composition herein remains in contact with the fabrics up to 24 hours, preferably from 1 minute to 12 hours and more preferably from 20 minutes to 6 hours. When used in its neat form the acidic liquid aqueous composition herein remains in contact with the fabrics up to 24 hours, preferably from 1 minute to 2 hours and more preferably from 1 minute to 1 hour.

[0032] The temperature of the bleaching process does have an influence on the stain removal performance and/or bleaching performance of the acidic liquid aqueous persulfate-containing compositions. More specifically, an increased temperature accelerates the bleaching process, i.e. diminishes the time required to bleach a given soil. Typically, the bleaching solutions occurring in the bleaching processes according to the present invention where the persulfate bleaching compositions as described herein are used in their diluted form have a temperature of from 4°C to 60°C, preferably from 10°C to 50°C and most preferably from 20°C to 40°C.

**[0033]** The process of bleaching fabrics according to the present invention further comprises as an essential step, the step of rinsing the fabrics with water after having been contacted with the acidic liquid aqueous persulfate-containing composition, before said composition has completely dried off.

[0034] The "bleaching step" in the process of bleaching fabrics of the present invention can be done by hand or in a washing-machine after the "alkaline step" that has been first carried out either in a hand washing operation or a washing machine operation. The "alkaline step" may optionally be followed by a rinsing step typically with water before the "bleaching step", i.e. before contacting the fabrics with the acidic liquid aqueous persulfate-containing composition.

**[0035]** For example when the "alkaline step" is carried out in a hand washing operation, the "bleaching step" may be performed in a hand operation, e.g. in soaking vessel outside the washing-machine, or in a washing machine by delivering the persulfate-containing composition via the detergent dispenser without any detergent or via the bleach dispenser if the machine is so provided.

[0036] When the "alkaline step" is carried out in a washing machine, the "bleaching step" may be performed by delivering the persulfate-containing composition via the detergent dispenser of said washing machine without any detergent, or via the bleach dispenser if the machine is so provided, or in a hand operation, e.g. in soaking vessel outside the washing-machine.

[0037] Surprisingly, the process of bleaching fabrics according to the present invention provides improved bleaching performance and improved overall stain removal performance, as compared to the same process without contacting the fabrics with the alkaline solution prior contacting them with the acidic liquid aqueous persulfate bleaching compositions or the same process where said alkaline step is performed after the step where the acidic liquid aqueous persulfate bleaching compositions has been contacted with the fabrics.

**[0038]** Indeed, effective bleaching performance is provided on a variety of soils, from hydrophobic to hydrophilic on both hydrophobic fabrics. Effective bleaching herein also includes effective whiteness.

**[0039]** Advantageously, the present invention provides not only effective bleaching performance but also good stain removal performance on various types of stains including greasy stains (e.g., lipstick, tomato, make-up) and/or enzymatic stains (e.g. blood, choco pudding).

[0040] It is speculated that it is the alkaline residualities left on the fabrics after the first alkaline step of the process of the bleaching fabrics according to the present invention that improves the bleaching performance and stain removal performance of the acidic liquid aqueous persulfate-composition contacted to the fabrics in a subsequent step both in its neat or diluted form. Indeed, these alkaline residualities activate the persulfate salt, e.g. potassium monopersulphate, and makes it bleaching action more effective.

**[0041]** An advantage of the persulfate compositions suitable for use in the process of bleaching fabrics according to the present invention is that they are physically and chemically stable upon prolonged periods of storage.

**[0042]** Chemical stability of these compositions may be evaluated by measuring the concentration of available oxygen at given storage time after having manufactured the compositions. By "chemically stable" it is meant herein that the compositions used herein comprising a persulfate salt or mixtures thereof and optionally an activator compound do not undergo more than 15% AvO loss, in one month at 25°C and preferably not more than 10%.

[0043] Available oxygen (AvO) loss of a persulfate containing composition can be measured by titration with potassium permanganate after reduction with a solution containing ammonium ferrous sulphate. Said stability test method is well known in the art and is reported, for example, on the technical information sheet of Curox<sup>R</sup> commercially available from Interox. Alternatively persulfate concentration can also be measured using a chromatography method described in the literature for peracids (F. Di Furia et al., Gas-liquid Chromatography Method for Determination of Peracids, Analyst, Vol 113, May 1988, p 793-795).

**[0044]** By "physically stable" it is meant herein that no phase separation occurs in the acidic liquid aqueous persulfate-containing compositions used herein for a period of 7 days at 50°C.

#### Optional ingredients

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**[0045]** The persulfate-containing compositions used in the process of bleaching fabrics according to the present invention after the so called "alkaline step" may comprise a variety of optional ingredients such as bleach activators, surfactants, chelating agents, radical scavengers, antioxidants, other stabilisers, builders, soil suspenders, polymeric soil release agents, catalysts, dye transfer agents, solvents, suds controlling agents, brighteners, perfumes, pigments, dyes and the like.

#### Bleach activators:

[0046] Suitable bleach activators for use in these acidic liquid aqueous persulfate-containing compositions include aldehyde activator compounds, ketone activator compounds, halide activator compounds and a mixture thereof. By "aldehyde", it is meant herein any compound which contains at least one carbonyl group and has two hydrogen atoms or carbon atom and a hydrogen atom attached directly to at least one of the carbonyl carbon atoms.

**[0047]** By "ketone", it is meant herein any compound which contains at least one carbonyl group and has two carbon atoms attached directly to at least one of the carbonyl carbon atoms.

[0048] These compounds can be aliphatic or aromatic substituted or unsubstituted, saturated or unsaturated, or acyclic, carbocyclic or heterocyclic.

[0049] By "halide", it is meant chloride, bromide and/or iodide.

[0050] These ketone, aldehyde and halide activator compounds react with persulfate salt to form an activated bleach-

ing specie, i.e. a dioxirane (when the activator compound is ketone or aldehyde) or a hypohalogenite (when the activator compound is a halide). These activation mechanisms are known in the art and are for example described in US 3822114.

[0051] Advantageously, when the acidic liquid aqueous persulfate-containing compositions used in the process of bleaching fabrics herein further comprise such a bleach activator, the bleaching performance and overall stain removal performance is further improved. It is the activated species, i.e. dioxirane and hypohalogenite, which are thought to greatly enhance the bleaching in the present invention over persulfate alone. These activation mechanisms mainly occur upon dilution of the compositions herein with water and are emphased with the pH jump that occurs upon this dilution in the neutral to alkaline pH range (i.e. around pH 6 and higher). It is at those pHs that more dioxiranes and/or hypohalogenites are generated thereby outstandingly improving the bleaching performance of the process of bleaching fabrics herein wherein after the "alkaline step" the acidic liquid aqueous persulfate-containing compositions are contacted to the fabrics in their diluted form.

**[0052]** Suitable aldehyde activator compounds for use herein include chloral-hydrate, acetaldehyde, butyraldehyde, benzaldehyde, and/or 4-trimethylammonio benzaldehyde methyl sulfate. Particularly preferred aldehyde activator compounds for use herein are acetaldehyde, butyraldehyde and/or, benzaldehyde and most preferred is acetaldehyde. Aldehyde activator compounds are in general less preferred than the ketone activators compounds as they may be oxidised during prolonged storage periods.

[0053] For example acetaldehyde may be commercially available from Aldrich.

[0054] Suitable aliphatic ketone activator compounds for use herein include acetacetone, 2,3-hexanedione, trimethylammonio acetone nitrate, 5-diethylbenzylammonio 2-pentanone nitrate, 5-diethylmethylammonio 2-pentanone nitrate, methyl pyruvate, diethyl keto malonate, 3-hydroxy-2-butanone, acetol, hexachloracetone, 2,5-hexanedione, phenylacetone, ethyl levulinate, 3-hydroxy-2-pentanone, acetone, 3-penten-2-one, methyl ethyl ketone, 4-hydroxy-3-methyl-2-butanone, 3-pentanone, and/or 2-heptanone.

[0055] For example acetacetone may be commercially available from Aldrich under its chemical name.

[0056] Suitable aromatic ketone activator compounds for use herein include hydroxyquinoline, 4-acetyl-1-methylpyridinium nitrate, di-2-pyridyl ketone N-oxide, 2-acetylquinoxaline, 2-acetyl-3-methylquinoxaline oxaline, di-2- pyridyl ketone, 6-acetyl-1,2,4-trimethyl quinolinium nitrate, 8-hydroxyquinoline N-oxide, methyl phenyl glyoxalate, N-methyl-pmorpholinio acetophenone methyl sulfate, 3-acetyl pyridine N-oxide, p-nitroacetophenone, m-nitroacetophenone, sodium p-acetyl benzene sulfonate, p-acetylbenzonitrile, 3,5-dinitroacetophenone, 4-trimethylammonioacetophenone nitrate, 4-methoxy-3-nitroacetophenone, p-chloroacetophenone, p-diacetylbenzene, N-methyl-p-morpholinio acetophenone nitrate, phenacyltriphenylphosphonium nitrate, 2-acetyl pyridine, 2-acetyl pyridine N-oxide, 3-acetyl pyridine, 4-acetyl pyridine, 4-acetyl pyridine N-oxide, 2,6- diacetyl pyridine, 3-acetyl pyridine N-oxide, and/or triacetylbenzene.

[0057] Suitable cyclic ketone activator compounds for use herein include cyclohexanone, 2-methylcyclohexanone, 2,6-dimethyl cyclohexanone, 3-methyl cyclohexanone, 4-ethyl cyclohexanone, 4-t-butyl cyclohexanone, 4,4-dimethyl cyclohexanone, methyl 4-oxo-cyclohexanone carboxylate, sodium 4-oxocyclohexanone carboxylate, 2-trimethylammoniocyclohexanone nitrate, 4-trimethylammonio cyclohexanone nitrate, 3 oxo-cyclohexyl acetic acid, cycloheptanone, 1,4-cyclohexadione, dehydrochloric acid, tropinone methonitrate, N-methyl-3-oxoquinuclidinium nitrate, cyclooctanone and/or cyclopentanone.

[0058] For example cyclohexanone may be commercially available from Aldrich under its chemical name.

[0059] Suitable heterocyclic ketone activator compounds for use herein include, 2,2,6,6-tetramethyl-4-piperidone hydrate, 1-methyl-4-piperidone N-oxide, N-carbethoxy 4-piperidone, tetrahydrothiopyran-4-one methonitrate, tetrahydrothiopyran-4-one S,S- dioxide, tetrahydrothiopyran-3-one,S,S,-dioxide, and/or 4-oxacyclohexanone.

[0060] All of the above-described aldehyde and ketone examples are all either commercially available or can obviously be synthesized by the skilled artisan having before him the teaching of the prior art Gardini et al., *J. Chem. Soc.(C)*, (1970) page 929 and Lyle et al., *J. Org. Chem.*, *Vol. 24* (March, 1959), page 342 are examples of such art and are hereby incorporated herein by reference. Also a method for the synthesis of oxopiperidinium compounds is described in "S.E. Denmark, D.C. Forbes, D.S. Hays, J.S. De Pue and R.G. Wilde, J.Org. Chem. 1995, 60, 1391-1407". [0061] Particularly preferred bleach activators for use herein are the ketone activator compounds and highly preferred are the ketones activator compounds bearing a positive charge.

[0062] Particularly suitable ketone activator compounds bearing a positive charge are for example oxopiperidinium salts having the following formula:

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wherein the carbonyl group >C=O, can be either at the 2, 3 or at the 4 position of the oxopiperidinium; R1 and R2 are each independently a substituted or unsubstituted hydrocarbon chain having from 1 to 20 carbon atoms, preferably a substituted or unsubstituted alkyl or alkenyl or alkinyl group containing from 1 to 20 carbon atoms, preferably from 1 to 12 carbon atoms, or a substituted or unsubstituted aryl group containing from 6 to 10 atoms, or a C1-C20 alkyl aryl group wherein the aryl group contains from 6 to 10 carbon atoms; X<sup>-</sup> can be any negative ion, e.g., triflate, tosilate, mesilate, nitrate, more preferred is triflate, mesilate, tosilate and most preferred is triflate and the oxopiperidinium ring can be mono or polysubstituted at the 2,3,5, or 6 positions by one or more substituents as defined for R1 or R2 above or a halogen atom.

[0063] Examples of oxopiperidinium salts particularly suitable for use herein include 1,1-dimethyl-3-oxopiperidinium nitrate, 1,1-dimethyl-4-oxopiperidinium triflate, 1,1-dimethyl-4-oxopiperidinium triflate, 1,1-dimethyl-4-oxopiperidinium nitrate, 1-benzyl-4-piperidone methonitrate, 1-t-butyl-1-methyl-4-oxopiperidinium nitrate, 1-(4-dodecylbenzyl) 1-methyl-4 oxopiperidinium chloride, 3-(N-methyl-4 oxopiperidinium)-propane sulfonate, 1-allyl-1-methyl-4-oxopiperidinium chloride, 1-methyl-1-(1-naphthyl-methyl)-4- oxopiperidinium chloride, 1-methyl-1-pentamethylbenzyl- 4-oxopiperidinium chloride, N,N'-dimethyl-N,N'-phenylene dimethylene -bis(4- oxopiperidinium nitrate), 1-benzyl-1-methyl-4-oxopiperidinium triflate, 1-benzyl-1-methyl-4-oxopiperidinium nitrate, 1-dodecyl-1-methyl-4-oxopiperidinium nitrate, 1 -octyl-1-methyl-4-oxopiperidinium nitrate, 1-nonyl-1-methyl-4-oxopiperidinium nitrate or a mixture thereof.

**[0064]** Other suitable ketone activator compounds bearing a positive charge suitable for use herein are for example the ammonium acetophenone salts according to the following formula:

$$R_3R_2R_1$$

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wherein the ammonium group can be either at the 2 , at the 3 position or at the 4 position; R1, R2 and R3 are each independently a substituted or unsubstituted hydrocarbon group having from 1 to 20 carbon atoms, preferably a substituted or unsubstituted alkyl, or alkenyl or alkinyl group containing from 1 to 20 carbon atoms, preferably from 1 to 12 carbon atoms, or a substituted or unsubstituted aryl group containing from 6 to 10 atoms or a C1-C20 alkyl aryl group wherein the aryl group contains from 6 to 10 carbon atoms; R4 is a substituted or nonsubstituted halogenate alkyl group having from 1 to 20 carbon atoms, preferably from 1 to 12 carbon atoms and more preferably is methyl, or trifluoromethyl or trichloromethyl or tribromomethyl, ;X can be any negative ion, e.g., triflate, tosilate, mesilate, or nitrate and the benzenic ring can be substituted at the 2, 3 and/or 4 positions by one or more substituents as defined for R1, R2 or R3 as described above or a halogen atom.

**[0065]** Examples of such ammonium acetophenone salts for use herein include 4-trimethylammonium acetophenone nitrate, 2-trimethylammoniumacetophenone mitrate, 4-triethylammoniumacetophenone mesilate, 3-trimethylammoniumacetophenone mesilate, trihalogenatedmethylphenyl ketone like trifluoromethylphenylketone, trichloromethylphenylketone, tribromomethylphenylketone or a mixture thereof.

**[0066]** Suitable halide activator compounds for use herein include chloride, bromide, iodide and mixtures thereof. Particularly preferred halide activator compounds for use herein is an alkali metal of chloride. For example sodium chloride may be commercially available from Aldrich under its chemical name.

[0067] Typically, the acidic liquid aqueous compositions suitable for use in the process of bleaching fabrics herein

comprise up to 10% by weight of the total composition of said ketone, aldehyde and/or halide activator compound, preferably from 0.05% to 5%, more preferably from 0.1% to 2% and most preferably from 0.2% to 1.5%.

#### Surfactants:

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**[0068]** The persulfate-containing compositions suitable for use in the process of bleaching fabrics herein may comprise a surfactant or a mixture thereof including nonionic surfactants, anionic surfactants, cationic surfactants, zwitterionic surfactants and/or amphoteric surfactants. Typically these surfactants do not contain functional groups that can be oxidised by the persulphate salt, or that can substantially hydrolize at a low pH.

**[0069]** Typically, the acidic liquid aqueous persulfate-containing compositions suitable for use in the process of bleaching fabrics herein may comprise from 0.01% to 50% by weight of the total composition of a surfactant or a mixture thereof, preferably from 0.1% to 30 % and more preferably from 0.2% to 10%.

[0070] Suitable anionic surfactants for use in the compositions herein include water-soluble salts or acids of the formula  $ROSO_3M$  wherein R preferably is a  $C_{10}$ - $C_{24}$  hydrocarbyl, preferably an alkyl or hydroxyalkyl having a  $C_{10}$ - $C_{20}$  alkyl component, more preferably a  $C_{12}$ - $C_{18}$  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 tetramethylammonium 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  $C_{12^-16}$  are preferred for lower wash temperatures (e.g., below about  $50^{\circ}C$ ) and  $C_{16^-18}$  alkyl chains are preferred for higher wash temperatures (e.g., above about  $50^{\circ}C$ ).

[0071] Other suitable anionic surfactants for use herein are water-soluble salts or acids of the formula  $RO(A)_mSO_3M$  wherein R is an unsubstituted  $C_{10}$ - $C_{24}$  alkyl or hydroxyalkyl group having a  $C_{10}$ - $C_{24}$  alkyl component, preferably a  $C_{12}$ - $C_{20}$  alkyl or hydroxyalkyl, more preferably  $C_{12}$ - $C_{18}$  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  $C_{12}$ - $C_{18}$  alkyl polyethoxylate (1.0) sulfate,  $C_{12}$ - $C_{18}$  alkyl polyethoxylate (2.25) sulfate,  $C_{12}$ - $C_{18}$  alkyl polyethoxylate (3.0) sulfate  $C_{12}$ - $C_{18}$  alkyl polyethoxylate (4.0) sulfate

[0072] Other particularly suitable anionic surfactants for use herein are alkyl sulphonates including water-soluble salts or acids of the formula  $RSO_3M$  wherein R is a  $C_6$ - $C_{22}$  linear or branched, saturated or unsaturated alkyl group, preferably a  $C_{12}$ - $C_{18}$  alkyl group and more preferably a  $C_{14}$ - $C_{16}$  alkyl group, 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).

[0073] Suitable alkyl aryl sulphonates for use herein include water- soluble salts or acids of the formula RSO<sub>3</sub>M wherein R is an aryl, preferably a benzyl, substituted by a C<sub>6</sub>-C<sub>22</sub> linear or branched saturated or unsaturated alkyl group, preferably a C<sub>12</sub>-C<sub>18</sub> alkyl group and more preferably a C<sub>14</sub>-C<sub>16</sub> alkyl group, and M is H or a cation, e.g., an alkali metal cation (e.g., sodium, potassium, lithium, calcium, magnesium etc) 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).

[0074] The alkylsulfonates and alkyl aryl sulphonates for use herein include primary and secondary alkylsulfonates and primary and secondary alkyl aryl sulphonates. By "secondary C6-C22 alkyl or C6-C22 alkyl aryl sulphonates", it is meant herein that in the formula as defined above, the SO3M or aryl-SO3M group is linked to a carbon atom of the alkyl chain being placed between two other carbons of the said alkyl chain (secondary carbon atom).

**[0075]** For example C14-C16 alkyl sulphonate salt is commercially available under the name Hostapur <sup>®</sup> SAS from Hoechst and C8-alkylsulphonate sodium salt is commercially available under the name Witconate NAS 8<sup>®</sup> from Witco SA. An example of commercially available alkyl aryl sulphonate is Lauryl aryl sulphonate from Su.Ma. Particularly preferred alkyl aryl sulphonates are alkyl benzene sulphonates commercially available under trade name Nansa<sup>®</sup> available from Albright&Wilson.

**[0076]** Other anionic surfactants useful for detersive 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 trieth-anolamine salts) of soap,  $C_8$ - $C_{24}$  olefinsulfonates, sulfonated polycarboxylic acids prepared by sulfonation of the pyro-

lyzed product of alkaline earth metal citrates, e.g., as described in British patent specification No. 1,082,179,  $C_8$ - $C_{24}$  alkylpolyglycolethersulfates (containing up to 10 moles of ethylene oxide); alkyl ester sulfonates such as  $C_{14-16}$  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  $C_{12}$ - $C_{18}$  monoesters) diesters of sulfosuccinate (especially saturated and unsaturated 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  $RC(CH_2CH_2O)_kCH_2COO$ -M+ wherein R is a  $C_{32}$ - $C_{32}$ -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).

[0077] Other particularly suitable anionic surfactants for use herein are alkyl carboxylates and alkyl alkoxycarboxylates having from 4 to 24 carbon atoms in the alkyl chain, preferably from 8 to 18 and more preferably from 8 to 16, wherein the alkoxy is propoxy and/or ethoxy and preferably is ethoxy at an alkoxylation degree of from 0.5 to 20, preferably from 5 to 15. Preferred alkylalkoxycarboxylate for use herein is sodium laureth 11 carboxylate (i.e.,  $RO(C_2H_4O)_{10}$ -CH $_2COONa$ , with R= C12-C14) commercially available under the name Akyposoft<sup>®</sup> 100NV from Kao Chemical Gbmh.

[0078] Suitable amphoteric surfactants for use herein include amine oxides having the following formula  $R_1$   $R_2R_3NO$  wherein each of R1, R2 and R3 is independently a saturated substituted or unsubstituted, linear or branched hydrocarbon chains of from 1 to 30 carbon atoms. Preferred amine oxide surfactants to be used according to the present invention are amine oxides having the following formula  $R_1$   $R_2R_3NO$  wherein R1 is an hydrocarbon chain comprising from 1 to 30 carbon atoms, preferably from 6 to 20, more preferably from 8 to 16, most preferably from 8 to 12, and wherein R2 and R3 are independently substituted or unsubstituted, linear or branched hydrocarbon chains comprising from 1 to 4 carbon atoms, preferably from 1 to 3 carbon atoms, and more preferably are methyl groups. R1 may be a saturated substituted or unsubstituted linear or branched hydrocarbon chain. Suitable amine oxides for use herein are for instance natural blend C8-C10 amine oxides as well as C12-C16 amine oxides commercially available from Hoechst.

**[0079]** Suitable zwitterionic surfactants for use herein contain both a cationic hydrophilic group, i.e., a quaternary ammonium group, and anionic hydrophilic group on the same molecule at a relatively wide range of pH's. The typical anionic hydrophilic groups are carboxylates and sulfonates, although other groups like sulfates, phosphonates, and the like can be used. A generic formula for the zwitterionic surfactants to be used herein is:

## $R_1-N^+(R_2)(R_3)R_4X^-$

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wherein  $R_1$  is a hydrophobic group;  $R_2$  is hydrogen,  $C_1$ - $C_6$  alkyl, hydroxy alkyl or other substituted  $C_1$ - $C_6$  alkyl group;  $R_3$  is  $C_1$ - $C_6$  alkyl, hydroxy alkyl or other substituted  $C_1$ - $C_6$  alkyl group which can also be joined to  $R_2$  to form ring structures with the N, or a  $C_1$ - $C_6$  carboxylic acid group or a  $C_1$ - $C_6$  sulfonate group;  $R_4$  is a moiety joining the cationic nitrogen atom to the hydrophilic group and is typically an alkylene, hydroxy alkylene, or polyalkoxy group containing from 1 to 10 carbon atoms; and X is the hydrophilic group which is a carboxylate or sulfonate group.

[0080] Preferred hydrophobic groups  $R_1$  are aliphatic or aromatic, saturated or unsaturated, substituted or unsubstituted hydrocarbon chains that can contain linking groups such as amido groups, ester groups. More preferred  $R_1$  is an alkyl group containing from 1 to 24 carbon atoms, preferably from 8 to 18, and more preferably from 10 to 16. These simple alkyl groups are preferred for cost and stability reasons. However, the hydrophobic group  $R_1$  can also be an amido radical of the formula  $R_a$ -C(O)-NH- $C(R_b)_2$ <sub>m</sub>, wherein  $R_a$  is an aliphatic or aromatic, saturated or unsaturated, substituted or unsubstituted hydrocarbon chain, preferably an alkyl group containing from 8 up to 20 carbon atoms, preferably up to 18, more preferably up to 16,  $R_b$  is selected from the group consisting of hydrogen and hydroxy groups, and m is from 1 to 4, preferably from 2 to 3, more preferably 3, with no more than one hydroxy group in any  $C(R_b)_2$  moiety. [0081] Preferred  $R_2$  is hydrogen, or a  $C_1$ - $C_3$  alkyl and more preferably methyl. Preferred  $R_3$  is a  $C_1$ - $C_4$  carboxylic acid group or C1-C4 sulfonate group, or a  $C_1$ - $C_3$  alkyl and more preferably methyl. Preferred  $R_4$  is  $C(CH2)_n$  wherein n is an integer from 1 to 10, preferably from 1 to 6, more preferably is from 1 to 3.

**[0082]** Some common examples of betaine/sulphobetaine are described in U.S. Pat. Nos. 2,082,275, 2,702,279 and 2,255,082, incorporated herein by reference.

**[0083]** Examples of particularly suitable alkyldimethyl betaines include coconut-dimethyl betaine, lauryl dimethyl betaine, decyl dimethyl betaine, 2-(N-decyl-N, N-dimethyl-ammonia)acetate, 2-(N-coco N, N-dimethylammonio) acetate, myristyl dimethyl betaine, palmityl dimethyl betaine, cetyl dimethyl betaine, stearyl dimethyl betaine. For example Coconut dimethyl betaine is commercially available from Seppic under the trade name of Amonyl 265<sup>®</sup>. Lauryl betaine is commercially available from Albright & Wilson under the trade name Empigen BB/L<sup>®</sup>.

**[0084]** Examples of amidobetaines include cocoamidoethylbetaine, cocoamidopropyl betaine or C10-C14 fatty acylamidopropylene(hydropropylene)sulfobetaine. For example C10-C14 fatty acylamidopropylene(hydropropylene)sulfobetaine is commercially available from Sherex Company under the trade name "Varion CAS<sup>®</sup> sulfobetaine".

[0085] A further example of betaine is Lauryl-immino-dipropionate commercially available from Rhone-Poulenc under the trade name Mirataine H2C-HA $^{\$}$ .

[0086] Suitable cationic surfactants for use herein include derivatives of quaternary ammonium, phosphonium, imidazolium and sulfonium compounds. Preferred cationic surfactants for use herein are quaternary ammonium compounds wherein one or two of the hydrocarbon groups linked to nitrogen are a saturated, linear or branched alkyl group of 6 to 30 carbon atoms, preferably of 10 to 25 carbon atoms, and more preferably of 12 to 20 carbon atoms, and wherein the other hydrocarbon groups (i.e. three when one hydrocarbon group is a long chain hydrocarbon group as mentioned hereinbefore or two when two hydrocarbon groups are long chain hydrocarbon groups as mentioned hereinbefore) linked to the nitrogen are independently substituted or unsubstituted, linear or branched, alkyl chain of from 1 to 4 carbon atoms, preferably of from 1 to 3 carbon atoms, and more preferably are methyl groups. Preferred quaternary ammonium compounds suitable for use herein are non-chloride/non halogen quaternary ammonium compounds. The counterion used in said quaternary ammonium compounds are compatible with any persulfate salt and are selected from the group of methyl sulfate, or methylsulfonate, and the like.

**[0087]** Particularly preferred for use in the compositions of the present invention are trimethyl quaternary ammonium compounds like myristyl trimethylsulfate, cetyl trimethylsulfate and/or tallow trimethylsulfate. Such trimethyl quaternary ammonium compounds are commercially available from Hoechst, or from Albright & Wilson under the trade name EMPIGEN CM<sup>®</sup>.

**[0088]** Amongst the nonionic surfactants, alkoxylated nonionic surfactants and especially ethoxylated nonionic surfactants are suitable for use herein. Particularly preferred nonionic surfactants for use herein are the capped alkoxylated nonionic surfactants as they have improved stability to the persulfate salts.

[0089] Suitable capped alkoxylated nonionic surfactants for use herein are according to the formula:

R<sub>1</sub>(O-CH<sub>2</sub>-CH<sub>2</sub>)<sub>n</sub>-(OR<sub>2</sub>)<sub>m</sub>-O-R<sub>3</sub>

wherein  $R_1$  is a  $C_8$ - $C_{24}$  linear or branched alkyl or alkenyl group, aryl group, alkaryl group, preferably  $R_1$  is a  $C_8$ - $C_{18}$  alkyl or alkenyl group, more preferably a  $C_{10}$ - $C_{15}$  alkyl or alkenyl group, even more preferably a  $C_{10}$ - $C_{15}$  alkyl group; wherein  $R_2$  is a  $C_1$ - $C_{10}$  linear or branched alkyl group, preferably a  $C_2$ - $C_{10}$  linear or branched alkyl group; wherein  $R_3$  is a  $C_1$ - $C_{10}$  alkyl or alkenyl group, preferably a  $C_1$ - $C_5$  alkyl group, more preferably methyl; and wherein n and m are integers independently ranging in the range of from 1 to 20, preferably from 1 to 10, more preferably from 1 to 5; or mixtures thereof.

**[0090]** These surfactants are commercially available from BASF under the trade name Plurafac<sup>®</sup>, from HOECHST under the trade name Genapol<sup>®</sup> or from ICI under the trade name Symperonic<sup>®</sup>. Preferred capped nonionic alkoxylated surfactants of the above formula are those commercially available under the tradename Genapol<sup>®</sup> L 2.5 NR from Hoechst, and Plurafac<sup>®</sup> from BASF.

#### **Chelating agents:**

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[0091] The acidic liquid aqueous persulfate-containing compositions suitable for use in the process of bleaching fabrics herein may comprise a chelating agent as a preferred optional ingredient. Suitable chelating agents may be any of those known to those skilled in the art such as the ones selected from the group comprising phosphonate chelating agents, amino carboxylate chelating agents, other carboxylate chelating agents, polyfunctionally-substituted aromatic chelating agents, ethylenediamine N,N'- disuccinic acids, or mixtures thereof.

**[0092]** The presence of chelating agents contribute to further enhance the chemical stability of the compositions. A chelating agent may be also desired in the compositions herein as it allows to increase the ionic strength of the compositions and thus their stain removal and bleaching performance on various surfaces.

[0093] Suitable phosphonate chelating agents for use herein may include alkali metal ethane 1-hydroxy diphosphonates (HEDP), alkylene poly (alkylene phosphonate), as well as amino phosphonate compounds, including amino aminotri(methylene phosphonic acid) (ATMP), nitrilo trimethylene phosphonates (NTP), ethylene diamine tetra methylene phosphonates, and diethylene triamine penta methylene phosphonates (DTPMP). 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 chelating agents for use herein are diethylene triamine penta methylene phosphonate (DTPMP) and ethane 1-hydroxy diphosphonate (HEDP). Such phosphonate cheating agents are commercially available from Monsanto under the trade name DEQUEST<sup>®</sup>.

[0094] 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 dihydroxy-

disulfobenzenes such as 1,2-dihydroxy -3,5-disulfobenzene.

[0095] 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'- disuccinic 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 tradename ssEDDS® from Palmer Research Laboratories.

[0096] Suitable amino carboxylates to be used herein include ethylene diamine tetra acetates, diethylene triamine pentaacetates, 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 diacetic 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 penta acetic acid, propylene diamine tetracetic acid (PDTA) which is, for instance, commercially available from BASF under the trade name Trilon FS® and methyl glycine di-acetic acid (MGDA).

[0097] Further carboxylate chelating agents to be used herein include salicylic acid, aspartic acid, glutamic acid, glycine, malonic acid or mixtures thereof.

[0098] Another chelating agent for use herein is of the formula:

$$R_1R_2R_3R_4$$
 $R_7$ 
 $R_8$ 
 $R_8$ 
 $R_8$ 
 $R_1R_2R_3R_4$ 
 $R_1R_2R_3R_4$ 

wherein  $R_1$ ,  $R_2$ ,  $R_3$ , and  $R_4$  are independently selected from the group consisting of -H, alkyl, alkoxy, aryl, aryloxy, -Cl, -Br, -NO<sub>2</sub>, -C(O)R', and - SO<sub>2</sub>R"; wherein R' is selected from the group consisting of -H, -OH, alkyl, alkoxy, aryl, and aryloxy; R" is selected from the group consisting of alkyl, alkoxy, aryl, and aryloxy; and  $R_5$ ,  $R_6$ ,  $R_7$ , and  $R_8$  are independently selected from the group consisting of -H and alkyl.

**[0099]** Particularly preferred chelating agents to be used herein are amino aminotri(methylene phosphonic acid), diethylene-triamino-pentaacetic acid, diethylene triamine penta methylene phosphonate, 1-hydroxy ethane diphosphonate, ethylenediamine N, N'-disuccinic acid, and mixtures thereof.

**[0100]** Typically, the acidic liquid aqueous persulfate-containing compositions suitable for use in the process of bleaching fabrics herein comprise up to 5% by weight of the total composition of a chelating agent, or mixtures thereof, preferably from 0.01% to 1.5% by weight and more preferably from 0.01% to 0.5%.

#### Radical scavengers:

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**[0101]** The persulfate-containing compositions suitable for use in the process of bleaching fabrics herein may comprise a radical scavenger or a mixture thereof.

[0102] Suitable radical scavengers for use herein include the well-known substituted mono and dihydroxy 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 anysole, 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. Such radical scavengers like N-propyl-gallate may be commercially available from Nipa Laboratories under the trade name Nipanox S1 <sup>®</sup>. Radical scavengers when used, are typically present herein in amounts ranging from up to 10% by weight of the total composition and preferably from 0.001% to 0.5% by weight.

**[0103]** The presence of radical scavengers may contribute to the chemical stability of the persulfate-containing bleaching compositions herein as well as to the safety profile of these compositions.

#### Test methods:

**[0104]** The bleaching performance may be evaluated by the following test methods on various type of bleachable stains.

[0105] A suitable test method for evaluating the bleaching performance on a soiled fabric according to the process of

bleaching of the present invention is the following: First a typical "alkaline step" is carried out either in a washing-machine (e.g., by introducing 225 g of a conventional detergent, e.g. Ariel future in the detergent dispenser of a typical washing machine, e.g., Sangiorgio Exacta 352ZX and running a washing cycle with the soiled fabrics) or in a hand soaking operation (e.g., by contacting soiled fabrics with a soaking solution typically obtained by diluting a conventional detergent, e.g., Ariel a mano and a dilution level of 140ml/10liters of water -typical soaking time is 1 minute to 20 minutes). Then an acidic liquid aqueous persulfate-containing composition as described herein is diluted with water typically at a dilution level of 1 to 100 ml/L, preferably 20 ml/L (composition :water), then the fabrics having previously undergone the "alkaline step" as described herein above, are soaked in it for 20 minutes to 6 hours and then rinsed. Alternatively the bleaching composition can be used in the washing machine at a dilution level of typically at a dilution level of 1 to 100 ml/L (composition :water). In the washing machine the soiled fabrics are bleached at a temperature of from 30° to 70°C for 10 to 100 minutes and then rinsed. Soiled fabrics/swatches with for example tea, coffee and the like may be commercially available from E.M.C. Co. Inc..

**[0106]** The bleaching performance is then evaluated by comparing side by side the soiled fabrics treated accordingly to the process where the "alkaline step" is before the "bleaching step" with those treated according to a different process, e.g., with the same compositions but with the "alkaline step" after the "bleaching step" or only the "bleaching step". A visual grading may be used to assign difference in panel units (psu) in a range from 0 to 4.

**[0107]** The stain removal performance may be evaluated with the same test method but with soiled fabrics soiled by different types of stains like enzymatic ones and/or greasy ones.

#### 20 Examples

**[0108]** Following liquid persulfate compositions were made by mixing the listed ingredients in the listed proportions (weight % unless otherwise specified). These compositions are suitable to be used in the "bleaching step" of the bleaching processes of the present invention.

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| Compositions (% weight)         | I          | II   | III  | IV   | ٧   |
|---------------------------------|------------|------|------|------|-----|
| Akyposoft 100 NV® 0.5           | 0.5        | -    | 0.3  | 0.4  |     |
| Curox <sup>®</sup>              | 3          | 2    | 4    | 4.3  | 5   |
| Trifluoromethylphenylketone 0.5 | -          | -    | 0.3  | -    |     |
| Acetoacetone                    | -          | 0.4  | -    | -    | 0.4 |
| HEDP                            | 0.1        | 0.05 | 0.16 | 0.1  | -   |
| внт                             | 0.05       | -    | -    | -    | 0.1 |
| Propyl gallate                  | -          | 0.1  | -    | 0.05 | -   |
| NaCl                            | -          | -    | 0.1  | 0.5  | -   |
| NaBr                            | -          | -    | -    | -    | 1.0 |
| Water and minors                | up to 100% |      |      |      | '   |
| Alkanizing agent up to pH       | 2          | 2.5  | 4    | 4    | 1.5 |

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| Compositions (% weight)                 | VI  | VII | VIII | IX  | Х   | ΧI  |
|---|-----|-----|------|-----|-----|-----|
| Akyposoft 100 NV®                       | 0.5 | 0.5 | 0.5  | 0.2 | 0.2 | -   |
| Curox <sup>®</sup>                      | 5   | 7   | 10   | 10  | 6   | 9.5 |
| Acetylacetone                           | 0.5 | 0.5 | 0.5  | -   | -   | -   |
| N,N dymethyl-4 -oxopiperidinium nitrate | -   | -   | -    | 0.5 | 0.5 | -   |

#### (continued)

| Compositions (% weight) | VI         | VII  | VIII | IX  | Х    | ΧI |
|-------------------------|------------|------|------|-----|------|----|
| Propyl gallate          | 0.05       | 0.05 | 0.1  | 0.1 | 0.05 | -  |
| Water and minors        | up to 100% |      |      |     |      | •  |
| рН                      | 2          | 1.8  | 1.5  | 1.5 | 2    | 2  |

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| Compositions (% weight)                 | XII        | XIII | XIV | χV  | XVI  | XVII |
|---|------------|------|-----|-----|------|------|
| Akyposoft 100 NV®                       | 0.5        | 0.5  | 0.5 | 0.2 | 0.2  | 0.2  |
| Curox <sup>®</sup>                      | 5          | 7    | 10  | 10  | 6    | 9.5  |
| Cyclohexanone                           | 0.5        | -    | 0.5 | 0.5 | -    | -    |
| N,N dymethyl-3 -oxopiperidinium nitrate | -          | 0.5  | -   | -   | 0.5  | -    |
| Propyl gallate                          | 0.05       | 0.05 | 0.1 | 0.1 | 0.05 | 0.1  |
| Water and minors                        | up to 100% |      |     |     |      |      |
| Hq                                      | 2          | 1.8  | 1.5 | 1.5 | 2    | 2    |

| Witconate NAS 8 (40% active)®            | 5          | 5    | 5   | 5   | 5    | -    |
|--|------------|------|-----|-----|------|------|
| Curox <sup>®</sup>                       | 9.5        | 9.5  | 9.5 | 9.5 | 9.5  | 9.5  |
| N,N dymethyl-4 -oxopiperidinium triflate | 0.5        | 1    | 0.1 | 0.5 | 0.5  | -    |
| HEDP                                     | 0.1        | 0.2  | 0.1 | -   | -    | 0.1  |
| внт                                      | 0.05       | 0.05 | 0.1 | 0.1 | 0.05 | 0.05 |
| Water and minors                         | up to 100% |      |     |     |      | 1    |
| pH                                       | 1.5        | 1.5  | 1.5 | 1.5 | 1.5  | 1.5  |

Compositions (% weight)

XVIII XIX XX XXI XXII XXIII

 ${f [0109]}$  Akyposoft 100 NV $^{f (B)}$  is a C12-C14 alkyl ethoxycarboxylate (EO10) commercially available from Kao Chemicals Gmbh.

45 BHT is di-tert butyl hydroxy toluene.

HEDP is ethane 1-hydroxy diphosphonate commercially available from Monsanto under the serie Dequest<sup>®</sup>. Witconate NAS 8 (40% active)<sup>®</sup> is C8 alkyl sulphonate surfactant.

Curox<sup>®</sup> is a triple salt of potassium monopersulfate, potassium sulphate and potassium bisulphate commercially available from Interox.

[0110] The following compositions are representative of the granular detergent compositions suitable to be diluted with water to provide an alkaline solution suitable to be used in the first step (alkaline step) of the process herein.

|     | Α   | В   | C    | D   |
|-----|-----|-----|------|-----|
| LAS | 8.0 | 8.0 | 5.25 | 4.8 |

## (continued)

|                             | A           | В     | С    | D     |
|-----------------------------|-------------|-------|------|-------|
| TAS                         | -           | -     | 1.25 | 1.6   |
| C25E3                       | 3.4         | 3.4   | -    | 1.2   |
| C45E7                       | -           | -     | 3.25 | 5.0   |
| QAS                         | -           | 0.8   | 0.8  | 2.0   |
| Zeolite A                   | 18.1        | 18.1  | -    | 19.5  |
| STPP                        | -           | -     | 19.7 | -     |
| NaSKS-6/citric acid (79:21) | -           | -     | -    | 10.6  |
| Carbonate                   | 13.0        | 13.0  | 6.1  | 21.4  |
| Bicarbonate                 | -           | -     | -    | 2.0   |
| Silicate                    | 1.4         | 1.4   | 6.8  | -     |
| Sulfate                     | 26.1        | 26.1  | 39.8 | 14.3  |
| PB4                         | 9.0         | 9.0   | 5.0  | 2.5   |
| TAED                        | 1.5         | 1.5.  | 0.5  | 0.25  |
| DETPMP                      | 0.25        | 0.25  | 0.25 | 0.2   |
| HEDP                        | 0.3         | 0.3   | -    | 0.3   |
| Enzymes                     | 0.003       | 0.004 | 0.01 | 0.015 |
| MA/AA                       | 0.3         | 0.3   | 0.8  | 1.6   |
| CMC                         | 0.2         | 0.2   | 0.2  | 0.4   |
| Photoactivated bleach (ppm) | 15          | 15    | 15   | 27    |
| Brightener                  | 0.09        | 0.09  | 0.08 | 0.19  |
| Perfume                     | 0.3         | 0.3   | 0.3  | 0.3   |
| Silicone antifoam           | 0.5         | 0.5   | 0.5  | 2.4   |
| Misc                        | c/minors to | 100%  | . '  | •     |
| Density in g/litre          | 850         | 850   | 750  | 750   |

| 5  |  |
|----|--|
| 10 |  |
| 15 |  |
| 20 |  |
| 25 |  |
| 30 |  |
| 35 |  |
| 40 |  |
| 45 |  |
| 50 |  |

| State   Section   Sectio |                                      | E     | F     | G     |
|--|--------------------------------------|-------|-------|-------|
| Sodium sulfate   | Blown Powder                         | l     | l     |       |
| LAS   3.0   3.0       DETPMP   0.4   0.5       CMC   0.4   0.4   0.5       Agglomerates  | Zeolite A                            | 15.0  | 15.0  | -     |
| DETPMP CMC CMC CMC O.4   | Sodium sulfate                       | 0.0   | 5.0   | -     |
| CMC MAVAA         0.4 d.0  | LAS                                  | 3.0   | 3.0   | -     |
| MA/AA         4.0         4.0         -           Agglomerates           C45AS         -         -         171.0           LAS         6.0         5.0         -           TAS         3.0         2.0         -           Silicate         4.0         4.0         -           Zeolite A         10.0         15.0         13.0           CMC         -         -         0.5           MA/AA         -         -         2.0           Carbonate         9.0         7.0         7.0           Spray On           Perfume         0.3         0.3         0.5           C45E7         4.0         4.0         4.0           4.0         4.0         4.0         4.0           C25E3         2.0         2.0         2.0           Dry additives    MA/AA   | DETPMP                               | 0.4   | 0.5   | -     |
| Agglomerates         C45AS         -         -         11.0           LAS         6.0         5.0         -           TAS         3.0         2.0         -           Silicate         4.0         4.0         -           Zeolite A         10.0         15.0         13.0           CMC         -         -         0.5           MA/AA         -         -         2.0           Carbonate         9.0         7.0         7.0           Spray On         Perfume         0.3         0.3         0.5           C45E7         4.0         4.0         4.0           4.0         4.0         4.0         4.0           C25E3         2.0         2.0         2.0           Dry additives         MA/AA         -         -         3.0           NaSKS-6         -         -         12.0           Citrate         10.0         -         8.0           Bicarbonate         7.0         3.0         5.0           PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.005         0.005           Lipase         0  | СМС                                  | 0.4   | 0.4   | -     |
| C45AS         -         -         11.0           LAS         6.0         5.0         -           TAS         3.0         2.0         -           Silicate         4.0         4.0         -           Zeolite A         10.0         15.0         13.0           CMC         -         -         0.5           MA/AA         -         -         2.0           Carbonate         9.0         7.0         7.0           Spray On           Perfume O.3         0.3         0.5         0.5           C45E7         4.0         4.0         4.0           4.0         2.0         2.0         2.0           Dry additives           MA/AA         -         -         3.0           NaSKS-6         -         -         12.0           Citrate         10.0         -         8.0           Bicarbonate         7.0         3.0         5.0           Carbonate         8.0         5.0         7.0           PVPVI/PVNO         0.5         0.5         0.5           Prote   | MA/AA                                | 4.0   | 4.0   | -     |
| LAS       6.0       5.0       -         TAS       3.0       2.0       -         Silicate       4.0       4.0       -         Zeolite A       10.0       15.0       13.0         CMC       -       -       0.5         MA/AA       -       -       2.0         Carbonate       9.0       7.0       7.0         Spray On         Perfume O.3       0.3       0.5         C45E7       4.0       4.0       4.0         4.0       2.0       2.0       2.0         Dry additives         MA/AA       -       -       3.0         NaSKS-6       -       -       12.0         Citrate       10.0       -       8.0         Bicarbonate       7.0       3.0       5.0         Carbonate       8.0       5.0       7.0         PVPVI/PVNO       0.5       0.5       0.5         Pectin degrading enzyme       0.02       0.005       0.005         Protease       0.026       0.016       0.047         Lipase       0.005       0.005       0.005         Amyl  | Agglomerates                         | L     | l .   |       |
| TAS         3.0         2.0         -           Silicate         4.0         4.0         -           Zeolite A         10.0         15.0         13.0           CMC         -         -         0.5           MA/AA         -         -         2.0           Carbonate         9.0         7.0         7.0           Spray On           Perfume         0.3         0.3         0.5           C45E7         4.0         4.0         4.0           4.0         2.0         2.0         2.0           Dry additives    MA/AA  | C45AS                                | -     | -     | 11.0  |
| Silicate         4.0         4.0         -           Zeolite A         10.0         15.0         13.0           CMC         -         -         0.5           MA/AA         -         -         2.0           Carbonate         9.0         7.0         7.0           Spray On           Perfume         0.3         0.3         0.5           C45E7         4.0         4.0         4.0           4.0         2.0         2.0         2.0           Dry additives           MA/AA         -         -         3.0           NaSKS-6         -         -         12.0           Citrate         10.0         -         8.0           Bicarbonate         7.0         3.0         5.0           Carbonate         8.0         5.0         7.0           PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.005         0.005           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0   | LAS                                  | 6.0   | 5.0   | -     |
| Zeolite A         10.0         15.0         13.0           CMC         -         -         0.5           MA/AA         -         -         2.0           Carbonate         9.0         7.0         7.0           Spray On           Perfume         0.3         0.3         0.5           C45E7         4.0         4.0         4.0           4.0         2.0         2.0         2.0           Dry additives    MA/AA  - NaSKS-6 12.0  NaSKS-6 12.0  Raditives  NaSKS-6 12.0  Raditives   | TAS                                  | 3.0   | 2.0   | -     |
| CMC         -         -         0.5           MA/AA         -         -         2.0           Spray On         7.0         7.0           Perfume Perfume C45E7 A.0   | Silicate                             | 4.0   | 4.0   | -     |
| MA/AA Carbonate         -         -         2.0           Spray On         7.0         7.0           Spray On           Perfume C45E7         0.3         0.3         0.5           C45E7         4.0         4.0         4.0           C25E3         2.0         2.0         2.0           Dry additives           MA/AA         -         -         3.0           NaSKS-6         -         -         12.0           Citrate         10.0         -         8.0           Bicarbonate         7.0         3.0         5.0           Carbonate         8.0         5.0         7.0           PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.05         0.02           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives  | Zeolite A                            | 10.0  | 15.0  | 13.0  |
| Carbonate         9.0         7.0         7.0           Spray On         Perfume         0.3         0.3         0.5           C45E7         4.0         4.0         4.0           C25E3         2.0         2.0         2.0           Dry additives           MA/AA         -         -         3.0           NaSKS-6         -         -         12.0           Citrate         10.0         -         8.0           Bicarbonate         7.0         3.0         5.0           Carbonate         8.0         5.0         7.0           PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.05         0.02           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.   | СМС                                  | -     | -     | 0.5   |
| Spray On         Perfume         0.3         0.3         0.5           C45E7         4.0         4.0         4.0           C25E3         2.0         2.0         2.0           Dry additives           MA/AA         -         -         3.0           NaSKS-6         -         -         12.0           Citrate         10.0         -         8.0           Bicarbonate         7.0         3.0         5.0           Carbonate         8.0         5.0         7.0           PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.005         0.02           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0  <   | MA/AA                                | -     | -     | 2.0   |
| Perfume         0.3         0.3         0.5           C45E7         4.0         4.0         4.0           C25E3         2.0         2.0         2.0           Dry additives           MA/AA         -         -         3.0           NaSKS-6         -         -         12.0           Citrate         10.0         -         8.0           Bicarbonate         7.0         3.0         5.0           Carbonate         8.0         5.0         7.0           PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.005         0.02           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0   | Carbonate                            | 9.0   | 7.0   | 7.0   |
| C45E7       4.0       4.0       4.0       4.0         C25E3       2.0       2.0       2.0         Dry additives         MA/AA       -       -       3.0         NaSKS-6       -       -       12.0         Citrate       10.0       -       8.0         Bicarbonate       7.0       3.0       5.0         Carbonate       8.0       5.0       7.0         PVPVI/PVNO       0.5       0.5       0.5         Pectin degrading enzyme       0.05       0.005       0.02         Protease       0.026       0.016       0.047         Lipase       0.009       0.009       0.009         Amylase       0.005       0.005       0.005         Cellulase       0.006       0.006       0.006         Silicone antifoam       5.0       5.0       5.0         Dry additives         Sodium sulfate       0.0       9.0       0.0         Balance (Moisture and Miscellaneous)       100.0       100.0       100.0   | Spray On                             |       |       |       |
| Dry additives         MA/AA         -         -         3.0           NaSKS-6         -         -         12.0           Citrate         10.0         -         8.0           Bicarbonate         7.0         3.0         5.0           Carbonate         8.0         5.0         7.0           PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.005         0.02           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0   | Perfume                              | 0.3   | 0.3   | 0.5   |
| Dry additives         MA/AA         -         -         3.0           NaSKS-6         -         -         12.0           Citrate         10.0         -         8.0           Bicarbonate         7.0         3.0         5.0           Carbonate         8.0         5.0         7.0           PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.005         0.02           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0   | C45E7                                | 4.0   | 4.0   | 4.0   |
| MA/AA         -         -         3.0           NaSKS-6         -         -         12.0           Citrate         10.0         -         8.0           Bicarbonate         7.0         3.0         5.0           Carbonate         8.0         5.0         7.0           PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.005         0.02           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0   | C25E3                                | 2.0   | 2.0   | 2.0   |
| NaSKS-6         -         -         12.0           Citrate         10.0         -         8.0           Bicarbonate         7.0         3.0         5.0           Carbonate         8.0         5.0         7.0           PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.005         0.02           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0   | Dry additives                        |       |       |       |
| Citrate         10.0         -         8.0           Bicarbonate         7.0         3.0         5.0           Carbonate         8.0         5.0         7.0           PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.005         0.02           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0  | MA/AA                                | -     | -     | 3.0   |
| Bicarbonate         7.0         3.0         5.0           Carbonate         8.0         5.0         7.0           PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.005         0.02           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0   | NaSKS-6                              | -     | -     | 12.0  |
| Carbonate         8.0         5.0         7.0           PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.005         0.002           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0  | Citrate                              | 10.0  | -     | 8.0   |
| PVPVI/PVNO         0.5         0.5         0.5           Pectin degrading enzyme         0.05         0.005         0.002           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0  | Bicarbonate                          | 7.0   | 3.0   | 5.0   |
| Pectin degrading enzyme         0.05         0.005         0.02           Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0  | Carbonate                            | 8.0   | 5.0   | 7.0   |
| Protease         0.026         0.016         0.047           Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0  | PVPVI/PVNO                           | 0.5   | 0.5   | 0.5   |
| Lipase         0.009         0.009         0.009           Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0   | Pectin degrading enzyme              | 0.05  | 0.005 | 0.02  |
| Amylase         0.005         0.005         0.005           Cellulase         0.006         0.006         0.006           Silicone antifoam         5.0         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0  | Protease                             | 0.026 | 0.016 | 0.047 |
| Cellulase Silicone antifoam         0.006         0.006         0.006           Dry additives         5.0         5.0         5.0           Sodium sulfate Balance (Moisture and Miscellaneous)         0.0         9.0         0.0           100.0         100.0         100.0         100.0  | Lipase                               | 0.009 | 0.009 | 0.009 |
| Silicone antifoam         5.0         5.0           Dry additives         Sodium sulfate         0.0         9.0         0.0           Balance (Moisture and Miscellaneous)         100.0         100.0         100.0  | Amylase                              | 0.005 | 0.005 | 0.005 |
| Dry additives  Sodium sulfate 0.0 9.0 0.0  Balance (Moisture and Miscellaneous) 100.0 100.0  | Cellulase                            | 0.006 | 0.006 | 0.006 |
| Sodium sulfate 0.0 9.0 0.0  Balance (Moisture and Miscellaneous) 100.0 100.0   | Silicone antifoam                    | 5.0   | 5.0   | 5.0   |
| Balance (Moisture and Miscellaneous) 100.0 100.0 100.0   | Dry additives                        |       |       |       |
|  | Sodium sulfate                       | 0.0   | 9.0   | 0.0   |
| Density (g/litre)   700   700   700  | Balance (Moisture and Miscellaneous) | 100.0 | 100.0 | 100.0 |
| ,  | Density (g/litre)                    | 700   | 700   | 700   |

The following compositions are representative of the liquid detergent compositions suitable to be used diluted with water to provide an alkaline solution suitable to be used in the first step of the process of bleaching fabrics according to the present invention.

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|    |  | Н    | I    | J    | К    | L    | М    | N    | 0    |
|----|--|------|------|------|------|------|------|------|------|
|    | LAS  | 10.0 | 13.0 | 9.0  | -    | 25.0 | -    | -    | -    |
| 10 | C25AS  | 4.0  | 1.0  | 2.0  | 10.0 | -    | 13.0 | 18.0 | 15.0 |
|    | C25E3S                                       | 1.0  | -    | -    | 3.0  | -    | 2.0  | 2.0  | 4.0  |
|    | C25E7  | 6.0  | 8.0  | 13.0 | 2.5  | -    | -    | 4.0  | 4.0  |
| 15 | TFAA   | -    | -    | -    | 4.5  | -    | 6.0  | 8.0  | 8.0  |
| 15 | QAS  | -    | -    | -    | -    | 3.0  | 1.0  | -    | -    |
|    | TPKFA  | 2.0  | -    | 13.0 | 2.0  | -    | 15.0 | 7.0  | 7.0  |
| 20 | Rapeseed fatty acids                         | -    | -    | -    | 5.0  | -    | -    | 4.0  | 4.0  |
|    | Citric                                       | 2.0  | 3.0  | 1.0  | 1.5  | 1.0  | 1.0  | 1.0  | 1.0  |
| 25 | Dodecenyl/tetra-<br>decenyl succinic<br>acid | 12.0 | 10.0 | -    | -    | 15.0 | -    | -    | -    |
|    | Oleic acid                                   | 4.0  | 2.0  | 1.0  | -    | 1.0  | -    | -    | -    |
|    | Ethanol                                      | 4.0  | 4.0  | 7.0  | 2.0  | 7.0  | 2.0  | 3.0  | 2.0  |
|    | 1,2 Propanediol                              | 4.0  | 4.0  | 2.0  | 7.0  | 6.0  | 8.0  | 10.0 | 13   |
| 30 | Mono Ethanol<br>Amine                        | -    | -    | -    | 5.0  | -    | -    | 9.0  | 9.0  |
|    | Tri Ethanol Amine                            | -    | -    | 8    | -    | -    | -    | -    | -    |
|    | NaOH (pH)                                    | 8.0  | 8.0  | 7.6  | 7.7  | 8.0  | 7.5  | 8.0  | 8.2  |
| 35 | Ethoxylated tetrae-<br>thylene pentamine     | 0.5  | -    | 0.5  | 0.2  | -    | -    | 0.4  | 0.3  |
|    | DETPMP                                       | 1.0  | 1.0  | 0.5  | 1.0  | 2.0  | 1.2  | 1.0  | -    |
| 40 | SRP 2  | 0.3  | -    | 0.3  | 0.1  | -    | -    | 0.2  | 0.1  |
| 40 | PVNO   | -    | -    | -    | -    | -    | -    | -    | 0.10 |
|    | Enzymes                                      | .007 | .009 | .009 | .009 | .01  | .013 | .011 | .015 |
|    | Boric acid                                   | 0.1  | 0.2  | -    | 2.0  | 1.0  | 1.5  | 2.5  | 2.5  |
| 45 | Na formate                                   | -    | -    | 1.0  | -    | -    | -    | -    | -    |
|    | Ca chloride                                  | -    | 0.01 | -    | 0.01 | -    | -    | -    | -    |
|    | Bentonite clay                               | -    | -    | -    | -    | 4.0  | 4.0  | -    | -    |
| 50 | Suspending clay<br>SD3                       | -    | -    | -    | -    | 0.6  | 0.3  | -    | -    |
|    | Balance Moisture and Miscellaneous           | 100  | 100  | 100  | 100  | 100  | 100  | 100  | 100  |

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 $LAS \hspace{1cm} \hbox{: Sodium linear $C_{12}$ alkyl benzene sulphonate} \\$ 

TAS : Sodium tallow alkyl sulphate

QAS :  $R_2.N^+(CH_3)_2(C_2H_4OH)$  with  $R_2 = C_{12}-C_{14}$ 

5 TFAA : C<sub>16</sub>-C<sub>18</sub> alkyl N-methyl glucamide.

TPKFA: C12-C14 topped whole cut fatty acids.

NaSKS-6 : Crystalline layered silicate of formula δ-Na<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>.

10 Carbonate

: Anhydrous sodium carbonate with a particle size between 200 μm and 900μm.

Bicarbonate : Anhydrous sodium bicarbonate with a particle size between 400 μm and 1200μm.

15 STPP : Anhydrous sodium tripolyphosphate

MA/AA : Copolymer of 1:4 maleic/acrylic acid, average molecular weight about 80,000

Zeolite A : Hydrated Sodium Aluminosilicate of formula Na<sub>12</sub>(A1O<sub>2</sub>SiO<sub>2</sub>)<sub>12</sub>.27H<sub>2</sub>O having a primary par-

ticle size in the range from 0.1 to 10 micrometers

Citrate : Tri-sodium citrate dihydrate of activity 86,4% with a particle size distribution between 425 µm

and 850 µm.

25 PB1 : Anhydrous sodium perborate monohydrate bleach, empirical formula NaBo2.H2O2

PB4 : Anhydrous sodium perborate tetrahydrate

TAED : Tetraacetyl ethylene diamine.

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Photoactivated Bleach : Sulfonated zinc phtlocyanine encapsulated in dextrin soluble polymer.

CMC : Sodium carboxymethyl cellulose.

35 HEDP : 1,1 -hydroxyethane diphosphonic acid.

DETPMP : Diethylene triamine penta (methylene phosphonic acid), marketed by Monsanto under the

Trade name Dequest 2060.

40 PVNO : Poly(4-vinylpyridine)-N-Oxide.

PVPVI : Poly (4-vinylpyridine)-N-oxide/copolymer of vinylimidazole and vinyl-pyrrolidone.

Silicone antifoam : Polydimethylsiloxane foam controller with siloxaneoxyalkylene copolymer as dispersing

agent with a ratio of said foam controller to said dispersing agent of 10:1 to 100:1.

SRP 2 : Diethoxylated poly (1,2 propylene terephtalate) short block polymer.

PPT : 10-propionic phenothiazine

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: Butyl syringate

Sulphate : Anhydrous sodium sulphate.

### Examples of processes of bleaching fabrics according to the present invention

[0111] The following processes of bleaching fabrics will illustrate the present invention:

[0112] Any of the combination "alkaline step"/"bleaching step" below are suitable, provided that the "alkaline step" is

before the "bleaching step".

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| In soaking vessel "Alkaline step" |                         |                       |       |  |  |  |  |
|-----------------------------------|-------------------------|-----------------------|-------|--|--|--|--|
|                                   | Alkaline detergent (ml) | Water volume (liters) | rinse |  |  |  |  |
|                                   | 50                      | 5L                    | no    |  |  |  |  |
|                                   | 100                     | 5L                    | yes   |  |  |  |  |
|                                   | 100                     | 10L                   | no    |  |  |  |  |
|                                   | 200                     | 10L                   | no    |  |  |  |  |

| In soaking vessel "Bleaching step" |                       |       |
|------------------------------------|-----------------------|-------|
| Persulfate composition (ml)        | Water volume (liters) | rinse |
| 50                                 | 5L                    | yes   |
| 100                                | 5L                    | yes   |
| 150                                | 10L                   | yes   |
| 200                                | 10L                   | yes   |

| In washing machine "Alkaline step" |                         |  |  |
|------------------------------------|-------------------------|--|--|
| Addition step                      | Alkaline detergent (ml) |  |  |
| in the main wash                   | 150                     |  |  |
| in the main wash                   | 150                     |  |  |
| in the main wash                   | 300                     |  |  |

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| In washing machine "Bleaching stop" |                             |  |
|-------------------------------------|-----------------------------|--|
| Addition step                       | Persulfate composition (ml) |  |
| during rinse cycle                  | 50                          |  |
| during rinse cycle                  | 100                         |  |
| during main wash *                  | 50                          |  |
| during main wash *                  | 100                         |  |

"during main wash\*" of the bleaching step it is to be understood that a washing cycle is run with the bleaching composition in absence of detergent composition, the bleaching composition being introduced directly in the detergent dispenser without the presence of the detergent or in the bleach dispenser if the machine is so provided.

**[0113]** The alkaline detergent used in the processes of bleaching fabrics as exemplified herein above can be any of the conventional detergent compositions available on the market, e.g. Ariel future, Dash future<sup>®</sup> when used in a washing machine, or Ariel a Mano<sup>®</sup> when used in a hand washing application. The persulfate containing compositions used in these processes can be any of the ones exemplified herein before, compositions I to XXIII.

[0114] Any washing machine may be used herein include European types as well as US types or Japanese types. An example is for instance San Giorgio Exacta 252 ZX.

**[0115]** All the above processes provide excellent bleaching performance as well as effective stain removal performance when bleaching fabrics as above indicated while being safe to the fabrics and colors.

#### **Claims**

1. A process of bleaching a fabric which comprises the steps of:

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- first contacting the fabric with an alkaline solution having a pH above 7,
- then contacting the fabric with an acidic liquid aqueous composition having a pH below 7 and comprising a persulfate salt, in its neat or diluted form,
- and subsequently rinsing the fabric with water.

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**2.** A process according to claim 1 wherein said alkaline solution has a pH of 8 to 13, preferably 8.5 to 12 and more preferably 9 to 11.5.

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3. A process according to any of the preceding claims wherein said alkaline solution is a liquid conventional detergent composition, in its neat or diluted form, or a conventional granular detergent composition diluted with water at a dilution level of up to 250 times its weight of water.

**4.** A process according to the preceding claims wherein said acid liquid aqueous composition comprises from 0.01% to 50% by weight of the total composition of a persulfate salt or mixtures thereof, preferably from 0.5% to 20%, more preferably from 1% to 15% and most preferably from 2% to 10%.

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**5.** A process according to any of the preceding claims wherein in said acid liquid aqueous composition said persulfate salt is a monopersulfate salt, preferably sodium monopersulfate and/or potassium monopersulfate.

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**6.** A process according to any of the preceding claims wherein said acidic liquid aqueous composition further comprises up to 10% by weight of the total composition of a ketone, aldehyde and/or halide activator compound, preferably from 0.05% to 5%, more preferably from 0.1% to 2% and most preferably from 0.2% to 1.5%.

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7. A process according to claim 6 wherein in the acidic liquid aqueous composition said activator is an oxopiperidinium salt, an ammonium acetophenone salt, acetacetone, 2,3-hexanedione, trimethylammonio acetone nitrate, 5diethylbenzylammonio 2-pentanone nitrate, 5-diethylmethylammonio 2-pentanone nitrate, methyl pyruvate, diethyl keto malonate, 3-hydroxy-2-butanone, acetol, hexachloracetone, 2,5-hexanedione, phenylacetone, ethyl levulinate, 3-hydroxy-2-pentanone, acetone, 3-penten-2-one, methyl ethyl ketone, 4-hydroxy-3-methyl-2-butanone, 3-pentanone, 2-heptanone, hydroxyquinoline, 4-acetyl-1-methylpyridinium nitrate, di-2-pyridyl ketone N-oxide, 2-acetylquinoxaline, 2-acetyl-3-methylquinoxaline oxaline, di-2- pyridyl ketone, 6-acetyl-1,2,4-trimethyl quinolinium nitrate, 8-hydroxyquinoline N-oxide, methyl phenyl glyoxalate, N-methyl-p-morpholinio acetophenone methyl sulfate, 3acetyl pyridine N-oxide, p-nitroacetophenone, m-nitroacetophenone, sodium p-acetyl benzene sulfonate, p-acetylbenzonitrile, 3,5-dinitroacetophenone, 4-trimethylammonioacetophenone nitrate, 4-methoxy-3-nitroacetophenone, p-chloroacetophenone, p-diacetylbenzene, N-methylp-morpholinio acetophenone nitrate, phenacyltriphenylphosphonium nitrate, 2-acetyl pyridine, 2-acetyl pyridine N-oxide, 3-acetyl pyridine, 4-acetyl pyridine, 4-acetyl pyridine N-oxide, 2,6- diacetyl pyridine, 3-acetyl pyridine N-oxide, triacetylbenzene, cyclohexanone, 2-methylcyclohexanone, 2,6-dimethyl cyclohexanone, 3-methyl cyclohexanone, 4-ethyl cyclohexanone, 4-t-butyl cyclohexanone, 4,4-dimethyl cyclohexanone, methyl 4-oxo-cyclohexanone carboxylate, sodium 4-oxo-cyclohexanone carboxylate, 2-trimethylammoniocyclohexanone nitrate, 4-trimethylammonio cyclohexanone nitrate, 3 oxo-cyclohexyl acetic acid, cycloheptanone, 1,4-cyclohexadione, dehydrochloric acid, tropinone methonitrate, N-methyl-3-oxoquinuclidinium nitrate, cyclooctanone, cyclopentanone, 2,2,6,6-tetramethyl-4-piperidone hydrate, 1-methyl-4-piperidone Noxide, N-carbethoxy 4-piperidone, tetrahydrothiopyran-4-one methonitrate, tetrahydrothiopyran-4-one S,S- dioxide, tetrahydrothiopyran-3-one, S, S, -dioxide, 4-oxacyclohexanone, or a mixture thereof.

**8.** A process according to any of the preceding claims 6 or 7 wherein in the acidic liquid aqueous composition said bleach activator is an oxopiperidinium salt having the following formula:

$$\begin{array}{c|c}
0 \\
\hline
3 & 4 & 5 \\
2 & 1 & 6 \\
N & + \\
R_1 & R_2
\end{array}$$

wherein the carbonyl group >C=O, can be either at the 2, 3 or at the 4 position of the oxopiperidinium; R1 and R2 are each independently a substituted or unsubstituted hydrocarbon chain having from 1 to 20 carbon atoms, preferably a substituted or unsubstituted alkyl or alkenyl or alkinyl group containing from 1 to 20 carbon atoms, preferably from 1 to 12, or a substituted or unsubstituted aryl group containing from 6 to 10 atoms, or a C1-C20 alkyl aryl group wherein the aryl group contains from 6 to 10 carbon atoms; X<sup>-</sup> can be any negative ion, e.g., triflate, tosilate, mesiltae, nitrate, more preferred is triflate, mesilate, tosilate and most preferred is triflate and the oxopiperidinium ring can be mono or polysubstituted at the 2,3,5, or 6 positions by one or more substituents as defined for R1 or R2 or a halogen atom and/or an ammonium acetophenone salt according to the following formula:

$$R_3R_2R_N$$

wherein the ammonium group can be either at the 2, at the 3 position or at the 4 position; R1, R2 and R3 are each independently a substituted or unsubstituted hydrocarbon group having from 1 to 20 carbon atoms, preferably a substituted or unsubstituted alkyl, or alkenyl or alkinyl group containing from 1 to 20 carbon atoms, preferably from 1 to 12, or a substituted or unsubstituted aryl group containing from 6 to 10 atoms or a C1-C20 alkyl aryl group wherein the aryl group contains from 6 to 10 carbon atoms; R4 is an alkyl group having from 1 to 20 carbon atoms,

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preferably from 1 to 12 and more preferably is methyl, or trifluoromethyl, trichloromethyl or tribromomethyl; X<sup>-</sup> can be any negative ion, e.g., triflate, tosilate, mesilate, or nitrate and the benzenic ring can be substituted at the 2, 3 and/or 4 positions by one or more substituents as defined for R1, R2 or R3 as described above or a halogen atom.

- 9. A process according to any of the preceding claims 6 to 8, wherein in the acidic liquid aqueous composition said bleach activator is 1,1-dimethyl-3-oxopiperidinium nitrate, 1,1-dimethyl-4-oxopiperidinium triflate, 1,1-dimethyl-3-oxopiperidinium triflate, 1,1-dimethyl-4-oxopiperidinium nitrate, 1-benzyl-4-piperidone methonitrate, 1-t-butyl-1-methyl-4-oxopiperidinium nitrate, 1-(4-dodecylbenzyl) 1-methyl-4 oxopiperidinium chloride, 3-(N-methyl-4 oxopiperidinium)-propane sulfonate, 1 -allyl-1-methyl-4-oxopiperidinium chloride, 1-methyl-1-(1-naphthyl-methyl)-4-oxopiperidinium chloride, 1-methyl-1-pentamethylbenzyl- 4-oxopiperidinium chloride, N,N'-dimethyl-N,N'-phenylene dimethylene bis(4- oxopiperidinlum nitrate), 1-benzyl-1-methyl-4-oxopiperidinium triflate, 1-benzyl-1-methyl-3-oxopiperidinium triflate, 1-benzyl-1-methyl-4-oxopiperidinium nitrate, 1-octyl-1-methyl-4-oxopiperidinium nitrate, 1-nonyl-1-methyl-4-oxopiperidinium nitrate, 4-trimethylammonium acetophenone nitrate, 2-trimethylammoniumacetophenone mesilate, trihalogenatedmethylphenyl ketone or a mixture thereof.
- **10.** A process according to any of the preceding claims wherein said acidic liquid aqueous composition has a pH in the range from 0.1 to 6, more preferably from 0.5 to 4, even more preferably from 1 to 3 and most preferably from 1 to 2.
- 11. A process according to any of the preceding claims wherein said acidic liquid aqueous composition further comprises at least an optional ingredient selected from the group consisting of chelating agents, radical scavengers, builders, surfactants, antioxidants, stabilisers, soil suspenders, polymeric soil release agents, catalysts, dye transfer agents, solvents, suds controlling agents, brighteners, perfumes, dyes, pigments and mixtures thereof. fabrics typically up to 24 hours, preferably from 5 minutes to 12 hours, and more preferably from 20 minutes to 6 hours.
- 13. A process of bleaching fabrics according to any of the preceding claims wherein the step of contacting the fabric with an alkaline solution having a pH above 7 is followed by a step where the resulting fabric is rinsed typically with water before being contacted with the acidic liquid aqueous composition comprising the persulfate salt.
- **14.** A process of bleaching fabrics according to any of the preceding claims wherein said acidic liquid aqueous composition in its neat form remains in contact with fabrics for a period of time sufficient to bleach said fabrics, typically from 1 minute to 12 hours, and more preferably from 1 minute to 1 hour.
- 15. A process of bleaching fabrics according to any of the preceding claims 1 to 13, wherein said acidic liquid aqueous composition is diluted with water at a dilution level up to 500 times its weight, preferably from 5 to 200 times and more preferably from 10 to 80 times and left to remains in contact with fabrics for a period of time sufficient to bleach said fabrics, typically up to 24 hours, preferably from 1 minute to 12 hours, more preferably from 20 minutes to 6 hours.



## **EUROPEAN SEARCH REPORT**

Application Number EP 97 87 0173

| Category                                 | Citation of document with indication of relevant passages  | on, where appropriate,  | Relevant<br>to claim  | CLASSIFICATION OF THE APPLICATION (Int.Cl.6) |
|--|--|---|---|--|
| A  | DE 44 27 662 A (PEROXIC<br>* page 3, line 22 - lir<br>1,3,10,18-23; examples   | ne 23; claims   | 1-6,10  | D06L3/02                                     |
| A  | EP 0 206 959 A (ATOCHEM * claims *   | 1)  | 1   |  |
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|  | The present search report has been o   | drawn up for all claims   |   |  |
|  | Place of search THE HAGUE  | Date of completion of the search  | Loi   | Examiner selet-Taisne, S                     |
| X : part<br>Y : part<br>doci<br>A : tech | ATEGORY OF CITED DOCUMENTS  icularly relevant if taken alone icularly relevant if combined with another ument of the same category inological background -written disclosure | E : earlier patent of after the filing of D : document cited L : document cited | iple underlying the<br>focument, but publicate<br>d in the application<br>d for other reasons<br>same patent famili | ished on, or                                 |