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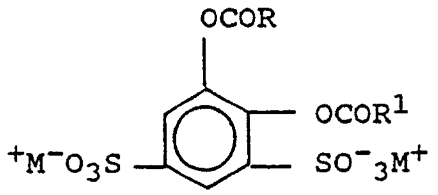
(54) Bleaching detergent compositions.

(57) Alkaline bleach detergent composition comprising a surface-active material, a detergency builder, a proteolytic enzyme, a peroxide compound and a bleach activator, characterized in that the bleach activator is a 1,2-diacyloxy benzene-3,5-disulphonate having the formula :



wherein R and R^1 are each individually a linear or non-linear alkyl group containing 1 to 10 carbon atoms, or a phenyl or substituted phenyl group containing 6 to 10 carbon atoms; and M is H, alkali metal, earth alkali metal, ammonium or substituted ammonium.

BLEACHING DETERGENT COMPOSITIONS

FIELD AND BACKGROUND

This invention relates to the field of bleaching detergent compositions, and in particular to the so-called low-temperature bleaching detergent compositions of the type wherein, during use, the bleach system functions by the generation of organic peroxyacids. Such compositions essentially comprise a peroxide compound, e.g. a persalt, such as sodium perborate, and an organic compound (the so-called "bleach activator" or "bleach precursor"), which in solution can react at relatively low temperatures with the peroxide compound to form an organic peroxyacid, which, unlike the peroxide compound, is effective in bleaching at lower temperatures, e.g. ≦ 60° C, such as are encountered in domestic washing and laundering operations.

Often such compositions, when used for fabric washing, also contain enzymes, especially proteolytic enzymes (proteases), which aid in the removal of proteolytic stains.

The term "peroxide compound" is used here to indicate those percompounds which liberate active oxygen in solution, particularly the inorganic peroxide compounds, such as the alkali metal perborates, persilicates, percarbonates and perphosphates. Bleaching detergent compositions containing such inorganic peroxide compounds and organic bleach activators therefor are known in the art. Various substances have been proposed in the art as bleach activators or precursors. Generally, these are carboxylic acid derivatives, e.g. esters (such as those described in GB Patent Specifications 836,988 and 970,950), acyl amides (such as those described in GB Patent Specifications 907,356; 855,735; 1,246,339 and US Patent Specification 4,128,494), and acyl azoles (such as those described in Canadian Patent Specification 844.481). Other, more recent patent publications disclosing bleach activators are, for example, US Patent 4,283,301; US Patent 4,412,934 and EP-A-0120591.

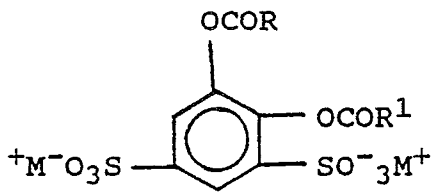
Typical bleach activators within the concept of the aforedescribed patent publications are N,N,N′,N′-tetraacetyl ethylene diamine (TAED), sodium acetoxy benzene sulphonate (SABS), sodium benzoyloxy benzene sulphonate (SBBS), sodium n- and iso-nonanoyloxy benzene sulphonate (SNOBS and iso-SNOBS).

With the current trend towards still lower fabric washing temperatures to, e.g., 40° C and below, there is a continuous incentive to further improve on the bleaching performance of fabric washing compositions without detrimentally affecting the enzyme action. There is also a continuous need of further developing new alternative formulations of fabric washing products that can meet the above requirements.

DESCRIPTION OF THE INVENTION

It has now been found that a certain class diacyloxy benzene sulphonates as defined hereinafter, when added to aqueous liquors containing a source of alkaline hydrogen peroxide, provide a very effective bleaching of oxidizable stains on fabrics at temperatures of ≦ 40° C. These diacyloxy benzene sulphonate compounds can be used as bleach activator in alkaline detergent compositions containing a peroxide compound and an enzyme to form low-temperature bleaching detergent composition which are quite effective for removing a wide class of stains from fabrics in domestic and laundering operations at or below 40° C.

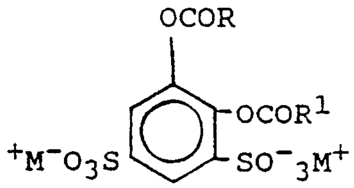
The bleach activator as used in the present invention is a 1,2-diacyloxy benzene-3,5-disulphonate having the formula:



wherein R and R¹ are each individually a linear or non-linear alkyl group containing 1 to 10 carbon atoms, or a phenyl or substituted phenyl group containing 6 to 10 carbon atoms; and M is H, alkali metal, earth alkali metal, ammonium or substituted ammonium.

These are novel compounds except disodium 1,2-dibenzoyloxy benzene-3,5-disulphonate, which is disclosed in German Patent Application N° 2,817,858 in admixture with sodium percarbonate as having antibacterial properties.

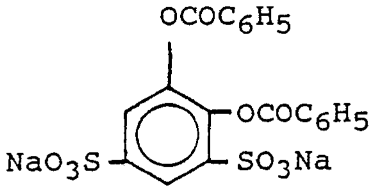
Accordingly, the invention provides an improved alkaline bleach detergent composition, especially for washing fabrics, comprising a surface-active material, a detergency builder, a proteolytic enzyme, a peroxide compound and a bleach activator, wherein the bleach activator is a 1,2-diacyloxy benzene-3,5-disulphonate having the formula :



wherein R and R' are each individually a linear or non-linear alkyl group containing 1 to 10 carbon atoms, or a phenyl or substituted phenyl group containing 6 to 10 carbon atoms; and M is H, alkali metal, earth alkali metal, ammonium or substituted ammonium.

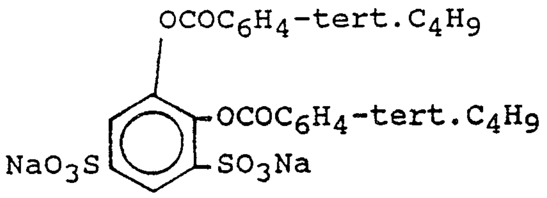
R and R' may be equal or different groups. Preferably, R and R' are equal. Preferred compounds are those wherein R and R' are phenyl, p-tert-butyl phenyl, or linear or non-linear C_6-C_8 alkyl groups, and wherein M is alkali metal, particularly sodium.

Thus, the following compounds are illustrative of bleach activators within this invention :



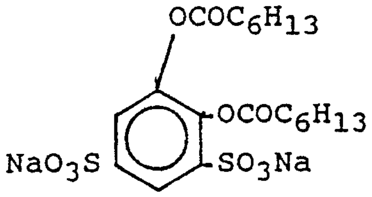
(I)

(disodium-1,2-dibenzoyloxy benzene-3,5 disulphonate)



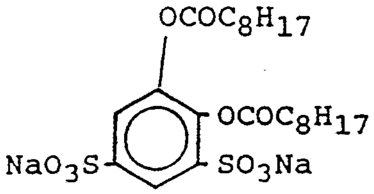
(II)

(disodium-1,2-di-tert.butyl benzoyloxy-3,5-disulphonate)



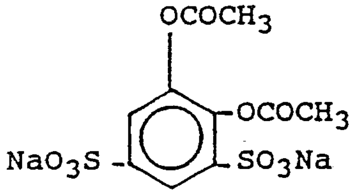
(III)

(disodium-1,2-diheptanoyloxy benzene-3,5-disulphonate)



(IV)

(disodium-1,2-dinonanoyloxy benzene-3,5-disulphonate)



(V)

(disodium-1,2-diacetoxy benzene-3,5-disulphonate)

The diacyloxy benzene disulphonates as defined herein are more weight-effective than the corresponding monoacyloxy benzene sulphonates, which means that, on an equal weight basis, they provide a much more effective bleaching, particularly at their optimal pH value. Similar isomeric compounds, such as the 1,3-diacyloxy benzene-4,6-disulphonates and the 1,3 diacyloxy benzene-4,5-disulphonates, are either less effective or difficult to synthesize, and are outside the purview of the present invention.

Each of the bleach activators as defined here can be used as a single compound in the bleaching detergent compositions of the invention, though mixtures of two or more compounds, e.g. (I) + IV) or (I) + (IV) + (V) or (II) + (V), may also be used, as desired. The use of such mixtures could be of benefit for the overal stain-removing properties and is also within the purview of the present invention.

Advantageously the alkaline detergent compositions of the invention comprising the bleach activator described herein will have a 2-5 g/l solution pH within the range of 8.5-10.5, with pH values of about 8.5-9.0 being optimal and preferred.

Peroxide compounds are well known in the art. They include the alkali metal peroxides, organic peroxide bleaching compounds such as urea peroxide, and inorganic persalt bleaching compounds, such as the alkali metal perborates, percarbonates, perphosphates and persulphates. Mixtures of two or more such compounds may also be suitable. Particularly preferred are sodium perborate tetrahydrate and, especially, sodium perborate monohydrate. Sodium perborate monohydrate is preferred because it has excellent storage stability while also dissolving very quickly in aqueous bleaching solutions.

Typically, the equivalent ratio of hydrogen peroxide (or a peroxide compound generating the equivalent amount of H_2O_2) to precursor will range from 0.5:1 to about 20:1, preferably 1:1 to 15:1, most preferably from 2:1 to 10:1.

A detergent formulation of the invention will normally also contain surface-active materials and detergency builders.

In the formulation of the invention, the bleach activator may be present at a level ranging from about 0.1% to 20% by weight, preferably from 0.5 to 10% by weight, particularly from 1% to 7.5% by weight, together with a peroxide bleaching compound, e.g. sodium perborate mono- or tetrahydrate, the amount of which is usually within the range of from about 2% to 40%, preferably from about 4% to 30%, particularly from about 10% to 25% by weight.

The surface-active material may be naturally derived or a synthetic material selected from anionic, nonionic, amphoteric, zwitterionic, cationic actives and mixtures thereof. Many suitable actives are commercially available and are fully described in literature, for example in "Surface Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch. The total level of the surface-active material may range up to 50% by weight, preferably being from about 1% to 40% by weight of the composition, most preferably 4% to 25%.

Synthetic anionic surface-active materials are usually water-soluble alkali metal salts of organic sulphates and sulphonates having alkyl radicals containing from about 8 to about 22 carbon atoms, the term alkyl being used to include the alkyl portion of higher aryl radicals.

Examples of suitable synthetic anionic detergent compounds are sodium and ammonium alkyl sulphates, especially those obtained by sulphating higher (C_8-C_{18}) alcohols produced, for example, from tallow or coconut oil; sodium and ammonium alkyl (C_9-C_{20}) benzene sulphonates, particularly sodium linear secondary alkyl (C_{10}-C_{15}) benzene sulphonates; sodium alkyl glyceryl ether sulphates, especially those esters of the higher alcohols derived from tallow or coconut oil and synthetic alcohols derived from petroleum; sodium coconut oil fatty acid monoglyceride sulphates and sulphonates; sodium and ammonium salts of sulphuric acid esters of higher (C_9-C_{18}) fatty alcohol alkylene oxide, particularly ethylene oxide, reaction products; the reaction products of fatty acids such as coconut fatty acids esterified with isethionic acid and neutralized with sodium hydroxide; sodium and ammonium salts of fatty acid amides of methyl taurine; alkane monosulphonates such as those derived by reacting alpha-olefins (C_8-C_{20}) with sodium bisulphite and those derived by reacting paraffins with SO_2 and Cl_2 and then hydrolyzing with a base to produce a random sulphonate; sodium and ammonium C_7-C_{12} dialkyl sulphosuccinates; and olefin sulphonates, which term is used to describe the material made by reacting olefins, particularly C_{10}-C_{20} alpha-olefins, with SO_3 and then neutralizing and hydrolyzing the reaction product. The preferred anionic detergent compounds are sodium (C_{11}-C_{15}) alkyl benzene sulphonates, sodium (C_{16}-C_{18}) alkyl sulphates and sodium (C_{16}-C_{18}) alkyl ether sulphates.

Examples of suitable nonionic surface-active compounds which may be used, preferably together with the anionic surface-active compounds, include in particular the reaction products of alkylene oxide, usually ethylene oxide, with alkyl (C_6-C_{22}) phenols, generally 5-25 EO, i.e. 5-25 units of ethylene oxides per molecule; the condensation products of aliphatic (C_8-C_{18}) primary or secondary linear or branched alcohols with ethylene oxide, generally 6-30 EO, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylene diamine. Other so-called nonionic surface-actives include alkyl polyglycosides, long chain tertiary amine oxides, long chain tertiary phosphine oxides and dialkyl sulphoxides.

Amounts of amphoteric or zwitterionic surface-active compounds can also be used in the compositions of the invention but this is not normally desired owing to their relatively high cost. If any amphoteric or zwitterionic detergent compounds are used, it is generally in small amounts in compositions based on the much more commonly used synthetic anionic and nonionic actives.

As stated above, soaps may also be incorporated in the compositions of the invention, preferably at a level of less than 25% by weight. They are particularly useful at low levels in binary (soap/anionic) or ternary mixtures together with nonionic or mixed synthetic anionic and nonionic compounds. Soaps which are used are preferably the sodium, or, less desirably, potassium salts of saturated or unsaturated C_{10}-C_{24} fatty acids or mixtures thereof. The amount of such soaps can be varied between about 0.5% and about 25% by weight, with lower amounts of about 0.5% to about 5% being generally sufficient for lather control. Amounts of soap between about 2% and about 20%, especially between about 5% and about 10%, are used to give a beneficial effect on detergency. This is particularly valuable in compositions used in hard water when the soap acts as a supplementary builder.

Detergency builder materials may be selected from 1) calcium sequestrant materials, 2) precipitating materials, 3) calcium ion-exchange materials and 4) mixtures thereof.

Examples of calcium sequestrant builder materials include alkali metal polyphosphates, such as sodium tripolyphosphate; nitrilotriacetic acid and its water-soluble salts; the akali metal salts of carboxymethyloxy succinic acid, ethylene diamine tetraacetic acid, oxydisuccinic acid, mellitic acid, benzene polycarboxylic acids, citric acid; and polyacetal carboxylates as disclosed in US patents 4,144,226 and 4,146,495.

Examples of precipitating builder materials include sodium orthophosphate, sodium carbonate and long chain fatty acid soaps.

Examples of calcium ion-exchange builder materials include the various types of water-insoluble crystalline or amorphous aluminosilicates, of which zeolites are the best known representatives, e.g. zeolites X, Y and A. In particular, the compositions of the invention may contain any one of the organic or inorganic builder materials, such as sodium or potassium tripolyphosphate, sodium or potassium pyrophosphate, sodium or potassium orthophosphate, sodium carbonate, the sodium salt of nitrilotriacetic acid, sodium citrate, carboxymethyl malonate, carboxymethyloxy succinate and the water-insoluble crystalline or amorphous aluminosilicate builder materials, or mixtures thereof.

These builder materials may be present at a level of, for example, from 5 to 80% by weight, preferably from 10 to 60% by weight.

The proteolytic enzymes which are suitable for use in the present invention are normally solid, catalytically active protein materials which degrade or alter protein types of stains when present as in fabric stains in a hydrolysis reaction. They may be made of any suitable origin, such as vegetable, animal, bacterial or yeast origin.

Proteolytic enzymes or proteases of various qualities and origins and having activity in various pH ranges of from 4-12 are available and can be used in the composition of the present invention. Examples of suitable proteolytic enzymes are the subtilisins, which are obtained from particular strains of B. subtilis and B. licheniformis, such as the commercially available subtilisins Maxatase® as supplied by Gist-Brocades N.V., Delft, Holland, and Alcalase®, as supplied by Novo Industri A/S, Copenhagen, Denmark.

Particularly suitable is a protease obtained from a strain of Bacillus having maximum activity throughout the pH range of 8-12, being commercially available, e.g. from Novo Industri A/S under the registered tradenames Esperase® and Savinase®. The preparation of these and analogous enzymes is described in British Patent Specification 1,243,784. Other commercial proteases are Kazusase® (obtainable from Showa-Denko of Japan), Optimase® (from Miles Kali-Chemie, Hannover, West-Germany), and Superase® (obtainable from Pfizer of USA).

The amount of proteolytic enzymes normally used in the composition of the invention may range from 0.001% to 10% by weight, preferably from 0.01% to 5% by weight, depending on their activity. They are generally incorporated in the form of granules, prills, or "marumes" in an amount such that the final washing product has a proteolytic activity of from about 2-20 Anson units per kilogram of final product.

Apart from the components already mentioned, the detergent compositions of the invention can contain any of the conventional additives in the amounts in which such materials are normally employed in fabric washing detergent compositions. Examples of these additives include lather boosters, such as alkanol amides, particularly the monoethanol amides derived from palmkernel fatty acids and coconut fatty acids; lather depressants, such as alkyl phosphates and silicones; anti-redeposition agents, such as sodium carboxymethyl cellulose and alkyl or substituted alkyl cellulose ethers; peroxide stabilizers, such as ethylene diamine tetraacetic acid, ethylene diamine tetra(methylene phosphonic acid) and diethylene triamine penta(methylene phosphonic acids, inorganic salts, such as sodium sulphate, and, usually present in very small amounts, fluorescent agents, perfumes, germicides, colourants and other enzymes, such as cellulases, lipases and amylases.

Other useful additives are polymeric materials, such as polyacrylic acid, polyethylene glycol and the copolymers (meth)acrylic acid and maleic acid, which may also be incorporated to function as auxiliary builders together with any of the principal detergency builders, such as the polyphosphates and especially aluminosilicates.

Specific examples of lipases are fungal lipases obtained from Humicola Lanuginosa or Thermomyces Lanuginosa, and bacterial lipases which react positively with the antibody of the lipase from Chromobacter Viscosum.

Fluorescent agents usable herein are, for example, those of. the diamino stilbene/cyanuric chloride types), e.g. Blankophor® MBBH; those of the distyryl benzene types, e.g. Tinopal® CBS and Tinopal® BLS; and those of the triazole types, e.g. Blankophor® BHC and Tinopal® RBS. A combination of Blankophor® BHC and Tinopal® BLS is preferred for cotton fabrics and Tinopal® RBS is preferred for nylon. Blankophor and Tinopal are registered Trade Marks.

The alkaline detergent compositions of the invention may be presented in any suitable state of aggregation, e.g. as solids or liquids. They are, however, preferably presented in free-flowing, particulate form, e.g. powdered or granular form, which can be produced by any of the known techniques commonly employed in the manufacture of such washing compositions, e.g. 1) by spray-drying an aqueous slurry comprising the surfactants and builders to form a detergent base powder, to which the heat-sensitive ingredients are added, such as, for example, the bleach activator, the enzyme(s), the peroxy compound, and optionally some other ingredients as conveniently desirable: 2) by dry-mixing or 3) by dry mixing/wet-blending. Generally, and for reasons of improving stability and handling, the bleach activator and enzymes are preferably added as granulated particles.

In a further specific embodiment the alkaline detergent composition of the invention is presented in the form of a non-aqueous, liquid detergent composition.

Example I

A detergent base powder of the following nominal composition was prepared by spray-drying.

Composition	parts by weight
Sodium alkyl benzene sulphonate	6.0
Fatty alcohol/7 ethoxylate	4.0
Alkaline sodium silicate (1:2)	6.0
Sodium triphosphate	36.0
Sodium carboxymethyl cellulose	0.3
Ethylene diamine tetraacetate (EDTA)	0.13
Fluorescent agent	0.3
Sodium sulphate	14.0

The following series of products were used in the washing tests:

Series I	% by weight			
	(A)	(B)	(C)	(D)
Detergent base	75.00	75.00	75.00	75.00
Ethylene diamine tetra-methylene phosphonic acid (Dequest ® 2041)	0.12	0.12	0.12	0.12
Sodium perborate monohydrate	14.00	14.00	14.00	14.00
Savinase ® 4.OT granules	1.00	1.00	1.00	1.00
Bleach activator :				
Compound (I)	3.83	-	-	-
SBBS	-	3.21	-	-
Iso-SNOBS	-	-	6.60	-
TAED	-	-	-	5.72
Water	to 100			
Series II	% by weight			
	(A)	(B)	(C)	(D)
Detergent base	75	75	75	75
Dequest ® 2041*	0.12	0.12	0.12	0.12
Sodium perborate monohydrate	1.46	1.07	1.96	5.02
Savinase ® 4.OT granules	1.00	1.00	1.00	1.00
Bleach activator :				
Compound (I)	3.83	-	-	-
SBBS	-	3.21	-	-
Iso-SNOBS	-	-	6.6	-
TAED	-	-	-	5.72
Water	to 100			

* Ethylene diamine tetra-(methylene phosphonic acid).

The bleach activator levels used in both series were precalculated to give 7 reflectance units (ΔR_{460}^*) on standard tea-stained test cloth in the series I formulation under the wash conditions as stated below, except that the pH was kept at 9.5.

The sodium perborate level of series I was kept constant at 14% by weight, whereas this level was varied in series II to maintain a constant 1:1 equivalent ratio of perborate to activator in the compositions A-D.

The washes were all carried out isothermally at 40° C in water of 15.FH in the presence of 4 g/l product. The wash pH was adjusted to pH 8.5 with sulphuric acid after adding all the wash components except the bleach activator.

The washes using standard tea-stained test cloths lasted 20 minutes, but those using RAGU® stain test cloths were limited to 10 minutes because the stain is deterged relatively easily.

Tea is a typical hydrophilic stain and RAGU® (a spaghetti sauce) is used as a typical hydrophobic stain.

Preparation of RAGU stain test cloth

To 40 ml of RAGU brand spaghetti sauce (available in the USA) add 4 ml of toluene and shake vigorously. Centrifuge for about 15 minutes, and carefully decant the clear red-orange supernatant toluene solution. Apply this to clean desized 10 x 15 cm cotton swatches in quantities of 150 µl per cloth, and allow the toluene to evaporate in the dark. Note :
This stain is bleached by both light and oxygen. It should therefore be used as quickly as possible after it has been made and it should be stored in the fridge wrapped in lightproof paper, and under nitrogen. Stains more than 7 days old should be discarded.

All washing experiments were repeated three times and averaged reflectance results are given in the following Table.

TABLE I

Results of Bleach/washing tests at 40 °C (ΔR460*)				
	I		II	
	Tea	RAGU	Tea	RAGU
(A)	11.1	17.1	5.2	19.3
(B)	11.5	11.2	3.3	11.5
(C)	7.8	12.6	2.7	9.0
(D)	6.0	10.7	3.7	8.9

The results clearly show superior low-temperature bleach performance of Products (A) according to the invention as compared with other products (B), (C) and (D) containing bleach activators of the art.

EXAMPLE II

A detergent base powder of the following nominal composition was prepared by spray-drying :

Composition	parts by weight
Sodium alkyl benzene sulphonate	6.0
Fatty alcohol/7 ethoxylate	4.0
Fatty alcohol/3 ethoxylate	3.0
Alkaline sodium silicate (1:2)	6.0
Sodium tripolyphosphate	25.0
EDTA	0.1
Sodium carboxymethyl cellulose	0.5
Sodium polyacrylate polymer	1.0
Minors (perfume, fluorescers, etc.)	1.5
Sodium sulphate (+ moisture) -- to	80.0 --

With this base powder, the following alkaline bleach detergent compositions were prepared and used in washing experiments.

Composition	(E)	(F)
	parts by weight	
Detergent base	80.0	80.0
Sodium perborate monohydrate	1.6	13.0
Bleach activator*	5.0	5.0
Dequest ® 2041	0.4	0.4
Savinase ® 4. OT granules	1.0	1.0

*The bleach activator used was compound (I). SBBS was used as comparison.

Two series of washing experiments were carried out with these compositions with and without bleach activator in a Tergotometer under isothermal wash conditions at 40°C in 18°FH water at product concentration of 5 gram/litre for 15 minutes on standard tea-stained, protein-stained and RAGU-stained test cloths. One series at wash pH 9.5 and another series at wash pH 8.5.

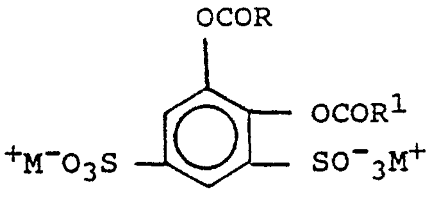
The results obtained are tabulated blow :

TABLE 2

At pH 9.5	Reflectance (ΔR_{460}^{*})					
System	Tea	Protein	RAGU			
(E) + compound (I)	5.4	25.3	30.0			
(E) - compound (I)	0.2	26.2	9.7			
At pH 8.5	Reflectance (ΔR_{460}^{*})					
	(E)			(F)		
System	Tea	Protein	RAGU	Tea	Protein	RAGU
+ compound (I)	6.0	26.7	38.5	5.9	25.6	16.2
+ SBBS	4.7	26.8	27.4	6.4	27.0	8.4
- activator	0.9	23.4	8.6	0.6	25.4	7.5

Claims

1. Alkaline bleach detergent composition comprising a surface-active material,.a detergency builder, a proteolytic enzyme, a peroxide compound and a bleach activator, characterized in that the bleach activator is a 1,2-diacyloxy benzene-3,5-disulphonate having the formula :



wherein R and R¹ are each individually a linear or non-linear alkyl group containing 1 to 10 carbon atoms, or a phenyl or substituted phenyl group containing 6 to 10 carbon atoms; and M is H, alkali metal, earth alkali metal, ammonium or substituted ammonium.

2. A bleach detergent composition according to Claim 1, characterized in that the bleach activator is disodium-1,2-dibenzoyloxy benzene-3,5-disulphonate.

3. A bleach detergent composition according to Claim 1, characterized in that the bleach activator is

disodium-1,2-di-tert. butyl benzoyloxy-3,5-disulphonate.

4. A bleach detergent composition according to Claim 1, characterized in that R and R_1 are linear or non-linear C_6-C_8 alkyl groups.

5. A bleach detergent composition according to any of the above Claims 1-4, characterized in that the composition contains from 2 to 40% by weight of the peroxide bleaching compound and from 0.1 to 20% by weight of the bleach activator in equivalent ratio of peroxide compound to bleach activator ranging from 0.5:1 to 20:1.

6. A bleach detergent composition according to any of the above Claims 1-5, characterized in that the composition has a solution pH at 2-5 g/l of 8.5-10.5.

7. A bleach detergent composition according to Claim 6, characterized in that said solution pH is from 8.5-9.0.

8. A bleach detergent composition according to any of the above Claims 1-7, characterized in that the composition has a proteolytic activity from 2-20 Anson units per kg of total product.