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(54) Bleaching composition.

(57) A stable and highly effective bleaching composition comprising (a) from 10% to 90%, preferably from 30% to 85% by weight of sodium percarbonate; (b) from 4% to 40%, preferably from 5% to 30% by weight of a bleach activator; and (c) from 5% to 85%, preferably from 10% to 70% by weight of an alkali metal bicarbonate, sesquicarbonate or dihydrogen orthophosphate, is disclosed. The composition can be used for admixing with a detergent composition or, preferably, for use as a bleach additive. Preferred compositions contain sodium bicarbonate and tetraacetyl ethylene diamine.

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BLEACHING COMPOSITION

This invention relates to a bleaching composition comprising essentially sodium percarbonate as the persalt and a bleach activator for said persalt, which composition can be used, as desired, for admixing with a detergent composition or, preferably, as a bleach additive to a wash liquor for improving the bleaching action of said wash liquor during the wash, particularly in the lower temperature region of from about 20 °C to 60 °C.

The term "bleach activator" used herein refers to the broad class of reactive organic compounds which in alkaline solutions containing a source of hydrogen peroxide, e.g. a persalt, will generate the corresponding peroxyacid, which, unlike the persalt or hydrogen peroxide, is an effective oxidising bleach at low temperatures. These compounds are therefore also referred to in the art as peroxyacid bleach precursors, of which N,N,N′,N′-tetraacetyl ethylene diamine (TAED), tetraacetyl glycoluril (TAGU), sodium acetoxybenzene sulphonate (SABS), sodium nonanoyloxybenzene sulphonate (SNOBS) and sodium benzoyloxybenzene sulphonate (SBOBS) are only a few typical examples in addition to the many other bleach precursor compounds described in literature, for example US Patents 1,246,339; 3,332,882; 4,128,494; 4,412,934; 4,675,393 and 4,751,015; GB Patent 1,382,594; EP-A-0185553, EP-A-170386, EP-A-0174132; EP-A-0120591; EP-A-0284292; EP-A-0303520; EP-A-0331229 and EP-A-0332294.

Sodium perborate mono- or tetrahydrate is currently the most widely used persalt bleaching agent in and with laundry detergent compositions. Bleaching compositions for use as a bleach additive comprising essentially sodium perborate mono- or tetrahydrate, TAED and urea have also been proposed and described in EP-A-0288170. These compositions containing sodium perborate are relatively stable and relatively easy to formulate. Sodium perborate, however, is a boron compound, which has been criticized on environmental grounds.

In a move to "greener" products, the invention makes use of the more environmentally acceptable persalt sodium carbonate perhydrate ($Na_2CO_3.1\frac{1}{2}\ H_2O_2$), commonly known as sodium percarbonate. Use of sodium percarbonate instead of sodium perborate, however, cannot be a matter of simple substitution. Unlike sodium perborate, sodium percarbonate is substantially less stable.

Japanese Patent Application N° 54-163906 discloses a detergent bleach composition comprising 20% by weight of sodium percarbonate, 40% by weight of heat-treated sodium bicarbonate and 3% by weight of a bleach activator. In order to improve stability it is essential, according to this reference, that 1) the sodium percarbonate used must have been subjected to a heat treatment at a temperature of about 70 °C-110 °C for 10-120 minutes, and that 2) the composition should contain a substantial amount of an acidic compound selected from succinic acid, citric acid and maleic acid.

Another important aspect of stability problems encountered with compositions containing higher levels of sodium percarbonate and a bleach activator is that which relates to the self-heating risk in factory handling, particularly if the product gets warm.

Various means have been proposed for stabilising or at least improving the stability of sodium percarbonate per se, for example by coating it with a borate-containing composition as described in GB Patent 2,123,404.

These so-called stabilised percarbonates, however, are still unsatisfactory in terms of self-heating risk when used in a bleaching composition comprising a bleach activator.

According to the invention there is now provided a stable and highly effective bleaching composition comprising essentially :

(a) from 10% to 90% by weight of sodium percarbonate;

(b) at least 4% to 40% by weight of a bleach activator; and

(c) from 5% to 85% by weight of an alkali metal bicarbonate, an alkali metal sesquicarbonate or an alkali metal dihydrogen orthophosphate,

wherein the ratio by weight of component (a) to (b) is not less than 4:5 and the ratio by weight of component (c) to (b) is not less than 5:4, with the proviso that, if component (b) is present at a level of more than 8%, the amount of component (c) should be at least 20%.

The sodium percarbonate used in the present invention need not have been subjected to a heat treatment.

Use of the so-called stabilised percarbonates in the composition of the invention is possible but appears to result only in a marginal improvement.

Hence, the great advantage of the present invention is that a satisfactorily stable bleaching composition can be formulated with untreated, substantially unstabilised sodium percarbonate as manufactured.

It is also of note that the present invention does not require the presence of acidic compounds in the

stated amounts as suggested in the above Japanese reference. Preferred compositions are therefore substantially free of succinic acid, citric acid or maleic acid.

The alkali metal salts used as component (c) are preferably the sodium salts, i.e. sodium bicarbonate, sodium sesquicarbonate and sodium dihydrogen orthophosphate ($NaH_2PO_4.2H_2O$), the sodium bi- or sesquicarbonates being particularly preferred as being non-phosphorus. Besides, the presence of sodium bicarbonate does not appear to influence the bleaching performance of the composition to any significant extent over the dosage range of interest, except for the dilution effect.

Apart from the above components, the bleaching composition of the invention may additionally contain sodium carbonate, as partial replacement for sodium bicarbonate. It has been found that sodium carbonate used at a level up to equal the amount of sodium bicarbonate does not affect the stability properties of the composition. Though not essential, the composition of the invention may further contain minor amounts of any known ingredients normally used in detergent or bleach compositions up to a level of about 10%, preferably not more than 5% by weight as desired, so long as their presence does not affect the stability properties of the composition.

Accordingly, in a preferred embodiment the composition is a fairly concentrated bleaching composition comprising essentially

a) from 30% to 85% by weight of sodium percarbonate;
b) from 5% to 30% by weight of a bleach activator;
c) from 10% to 70% by weight of sodium bicarbonate; and
d) from 0% to 35% by weight of sodium carbonate.

To this composition, for example, about 1% by weight of coloured detergent base powder speckles may be added for the purpose of product identification. Preferred speckles are, for example, blue- or green-coloured sodium carbonate particles.

specifically preferred are those compositions which combine optimal stability with optimal performance when used as a bleach additive, comprising essentially :

(a) from 40% to 60% by weight of sodium percarbonate;
(b) from 12% to 25% by weight of bleach activator;
(c) from 20% to 50% by weight of sodium bicarbonate; and
(d) from 0% to 15% by weight of sodium carbonate.

The bleach activator

As explained before, any bleach activator from the broad class of peroxyacid bleach precursors can be used in the composition of the invention. One preferred bleach activator is TAED because it is relatively harmless and is commercially available. Other preferred bleach activators are SNOBS; SBOBS; the quaternary ammonium-substituted peroxyacid bleach precursors as described in US Patent 4,751,015, EP-A-0331229, EP-A-0284292 and EP-A-303,520; and 2-methyl-4H,3,1-benzoxazin-4-one as described in EP-A-0332294, because of their high technical performances.

These activators can be used in their normal crystalline forms or, preferably, in the form of agglomerates or granulated particles having a particle size from 0.2 to 2.0 mm made from fine crystalline peroxyacid bleach precursor material of a particle size preferably below 150 μm. If the bleach activator is used in the unprotected crystalline form, which is possible under very dry conditions, it should preferably be present as coarse crystals having a size of not less than 0.2 mm. Still, agglomerates or granulated particles of bleach activators are the preferred forms for use in the present invention. They minimise direct contact between the sodium percarbonate and the bleach activator, thereby preventing undue perhydrolysis reactions from occuring during storage. The technique of agglomeration and granulation is well known in the art, and any of these techniques can be used for preparing the bleach activator granules. For example, bleach activator granules as described in EP-A-0240057 and EP-A-0241962 are very suitable for use in the present invention.

The sodium percarbonate

Any grade of sodium percarbonate can be used without any prior treatment being necessary, but a coarse grade is also preferable here. A highly suitable percarbonate grade is that of which at least 60% by weight has a particle diameter larger than 250 μm. Other components used in the compositions will preferably also be of similar coarse grades.

The bleaching composition of the invention can be prepared by simple mixing of the dry ingredients in any suitable mixing equipment.

EXAMPLE I

The following bleach compositions were prepared by dry mixing sodium percarbonate, TAED granules * and coarse sodium bicarbonate (mean particle size > 125 μm). The compositions in thermos-flasks were put in an oven at 100° C for about 24 hours, wherein the temperature of the compositions is monitored. The results of this test, which was designed to assess the self-heating risk, are shown in the following Table :

TABLE 1

Composition of the invention Percarbonate/TAED granule/bicarbonate (wt.%)	Max. rate of temp. rise (° C/minute)
1) 90/5/5	41
2) 85/5/10	13
3) 68/12/20	51
4) 64/14/22	7
5) 60/15/25	13
6) 59.5/10.5/30	8
7) 53/18/29	11.5
8) 52.5/17.5/30	27
9) 51/9/40	18
10) 40/30/30	16.5
11) 40/20/40	12.5
12) 30/30/40	6.5
13) 30/20/50	7.5
Compositions outside the invention	**Max. rate of temp. rise (° C/minute)**
14) 95/5/0	125
15) 90/10/0	>250
16) 85/15/0	167
17) 85.5/9.5/5	79
18) 80/20/0	>250
19) 80/10/10	109
20) 76.5/13.5/10	89
21) 75/25/0	189
22) 75/10/15	143
23) 70/15/15	>250
24) 67.5/22.5/10	219
25) 50/30/20	167
26) 40/40/20	219

* TAED granule composition :	% by weight
TAED	83.0
Sodium sulphate	9.2
Sokalan ® CP5-polymer	2.3
Bentonite clay	2.3
Water + minor salts	3.2

From the above results it is clear that the compositions of the invention show relatively low rates of temperature rise as compared with the compositions outside the invention showing relatively high to very high rates of temperature rise, which is indicative of the excellent stability of the compositions of the invention in contrast with the compositions outside the invention showing high to very high rate of exothermic decomposition.

EXAMPLE II

68/12/20 Formulations were prepared with 20% of various diluent materials. These formulations were subjected to the same exothermic decomposition testing at 100 °C as in Example I. The results are tabulated below.

TABLE 2

Composition (68/12/20)	Max. rate of temp. rise (°C/min.
1) 20% $NaH_2PO_4.2H_2O$	29
2) 20% $NaH.CO_3/Na_2CO_3$	40
3) 20% $Na_5P_3O_{10}$	146
4) 20% Na_2SO_4	>250
5) 20% Na_2CO_3	>250
6) 20% Citric acid	125
7) 20% K_2CO_3	>250
8) 20% Na_2HPO_4	225

The above results again show the superior stability of compositions 1) and 2) within the invention as compared with compositions 3) to 8) outside the invention.

EXAMPLE III

This Example demonstrates the effectiveness of a bleach additive according to the invention in a washing system using a main wash powder and a builder additive. The bleach additive had the following formulation :

Sodium percarbonate	52.5% by weight
Sodium bicarbonate	29.0% by weight
TAED granules (83% wt active)	17.5% by weight
Blue speckles (of main wash powder)	1.0% by weight
	100.0% by weight

Using a Tergotometer with a heat up to 60 ° C, detergencies with and without bleach additive were compared at the extremes of water hardness using two bleach-sensitive test cloths.

The main wash powder composition (spray-dried)	wt.%
Linear alkylbenzene sulphonate	14.2
Nonionic surfactant	6.2
Zeolite (anhydrous)	37.7
Sodium silicate	2.2
Sodium carbonate	16.8
Acrylic/maleic copolymer	6.2
Sodium carboxymethyl cellulose	0.8
Propteolytic enzyme	0.6
Anti-foam ingredients	1.1
Minor ingredients, salts, moisture	14.2
	100.0
Builder additive composition (dry-mixed)	wt.%
Zeolite A (anhydrous basis)	58.82
Sodium carbonate	17.9
Sodium sulphate	2.23
Sodium hydroxide	0.38
Sodium citrate	5.0
Nonionic surfactant	2.0
Sodium carboxymethyl cellulose	1.54
Perfume	0.1
Water	12.32

The results were as shown in the following Table 3.

TABLE 3

Conditions		
Water hardness		
French (°FH)	7	46
German (°DH)	4	24
Main wash powder dosage (g/l)	4	4
Builder additive dosage (g/l)	0	3
Bleach additive dosage (g/l)	0, 1 or 2	0, 1 or 2
Reflectance increase (ΔR_{460*})		
Test cloth 1 (tea on cotton) :		
Without bleach	1.51	3.41
with 1 g/l	15.41	20.11
with 2 g/l	17.61	25.51
Test cloth 2 (red wine on cotton) :		
Without bleach	15.46	14.96
with 1 g/l	35.16	39.46
with 2 g/l	35.06	41.06

EXAMPLE IV

This Example demonstrates the benefit for the use of a bleach additive according to the invention in a washing process over washing with commercial bleaching detergent compositions.

The experiments were carried out, using the 60° C cycle of a Miele (Trade Mark) 756 washing machine, in 30° (French) hard water, with a 2.5 kg load of clean cotton sheeting and terry towelling. Detergency was monitored, using two different blood-stained test cloths.

Three washes were carried out, using detergent powders as follows :

EXAMPLE IV

Pre-wash :	Main wash powder (Example III)	55 g
	Builder additive (Example III)	20 g
Main wash :	Main wash powder (Example III)	100 g
	Builder additive (Example III)	20 g
	Bleach additive (Example III)	45 g
Comparative Example A :		
No pre-wash		
Main wash :	Commercial bleaching detergent powder A containing sodium perborate + TAED	210 g
Comparative Example B :		
Pre-wash :	Commercial bleaching detergent powder B containing sodium perborate + TAED	112 g
Main wash :	Commercial bleaching detergent powder B containing sodium perborate + TAED	140 g

Detergency results (reflectance increases, ΔR_{460*}) were as follows :

	IV	A	B
Test cloth 3 (non-clotting blood on cotton)	63.09	36.10	43.82
Test cloth 4 (blood/milk/ink on cotton)	33.91	21.90	18.59

Claims

1. Stable bleaching composition comprising a persalt and a bleach activator, characterised in that it comprises essentially :
(a) from 10% to 90% by weight of sodium percarbonate;
(b) from 4% to 40% by weight of a bleach activator; and
(c) from 5% to 85% by weight of an alkali metal bicarbonate, an alkali metal sesquicarbonate or an alkali metal dihydrogen orthophosphate,
wherein the ratio by weight of component (a) to (b) is not less than 4:5 and the ratio by weight of component (c) to (b) is not less than 5:4, with the proviso that, if component (b) is present at a level of more than 8%, the amount of component (c) should be at least 20%.

2. Bleaching composition according to Claim 1, characterised in that the sodium percarbonate has not been subjected to heat treatment.

3. Bleaching composition according to Claim 1 or 2, characterised in that the composition is substantially free of succinic acid, citric acid or maleic acid.

4. Bleaching composition according to Claim 1, 2 or 3, characterised in that it comprises essentially :
a) from 30% to 85% by weight of sodium percarbonate;
b) from 5% to 30% by weight of a bleach activator;
c) from 10% to 70% by weight of sodium bicarbonate; and
d) from 0% to 35% by weight of sodium carbonate.

5. Bleaching composition according to Claim 4, characterised in that it comprises essentially :
(a) from 40% to 60% by weight of sodium percarbonate;
(b) from 12% to 25% by weight of bleach activator;
(c) from 20% to 50% by weight of sodium bicarbonate; and
(d) from 0% to 15% by weight of sodium carbonate.

6. Bleaching composition according to any of the above Claims 1-5, characterised in that the bleach activator is a peroxyacid bleach precursor.

7. Bleaching composition according to Claim 6, characterised in that said peroxyacid bleach precursor is N,N,N′,N′-tetraacetyl ethylene diamine (TAED).