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(54) Laundry compositions containing peroxyacid bleach and perfume particles.

(57) Laundry detergent or bleaching compositions containing peroxyacid bleach and perfume particles formed by adsorbing a perfume composition onto silica particles. The particles protect the perfume from oxidation by the bleach allowing for more efficient delivery of perfume to the laundry process and fabrics.

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LAUNDRY COMPOSITIONS CONTAINING PEROXYACID BLEACH AND PERFUME PARTICLES

FIELD OF THE INVENTION

The invention pertains to granular laundry detergent or bleaching compositions comprising a peroxyacid bleach and perfumed silica particles. The particles protect the perfume from oxidation by the bleach during storage and use of the composition. The invention thereby provides improved product odor, odor during the laundry process, and delivery of perfume to fabrics. The perfume particles are preferably admixed with detergent or bleach granules to provide finished granular detergent or bleach compositions containing peroxyacid bleaches.

BACKGROUND OF THE INVENTION

Perfumes are a desirable part of the laundry process. They are used to cover up the chemical odors of the cleaning ingredients and provide an aesthetic benefit to the wash process and, preferably, the cleaned fabrics. Perfumes are often added directly to laundry compositions, such as by spraying the perfume onto finished compositions. However, perfumes are, in general, volatile and many perfume ingredients can be destroyed or damaged by contact with cleaning ingredients, especially alkali and bleaches. To minimize direct contact between perfume and bleach components in laundry compositions, bleaches are sometimes admixed after perfume spray-on. Even this does not avoid oxidation of perfumes by bleaches, particularly when reactive bleaches such as peroxyacids are present.

One solution to this incompatibility problem is encapsulation of the perfume. This increases the expense and complexity of formulation and does not always provide sufficient protection.

It is known in the food industry to put flavors onto silica gel particles to form dry, flowable flavor powders. Flavor oil to silica gel ratios of up to 3: 1 can be used. When the particles are added to water, the flavor is released. However the art has not recognized that silica particles can be used to protect perfumes from peroxyacid bleaches present in laundry compositions.

SUMMARY OF THE INVENTION

The present invention is directed to granular laundry detergent or bleaching compositions containing a peroxyacid bleach and perfume particles in which the perfume is adsorbed onto certain silica particles. The silica particles are preferably incorporated into finished laundry detergent or bleaching compositions containing peroxyacid bleach, although the perfume particles and bleach can be used as is, such as in a laundry booster product. The silica particles have a diameter of from about 0.001 micron to about 15 microns and are present at a level to provide from about 0.001% to about 5% perfume by weight in the laundry detergent or bleaching compositions.

In addition to separating and protecting the perfume from the bleach, it is believed that the silica particles deposit on fabrics and enhance delivery of the perfume to the fabrics. The use of the dry flowable perfume particles herein in laundry compositions containing peroxyacid bleach can thus be a cost effective and efficient way to deliver perfume during the laundry process and to fabric. In addition, it provides greater flexibility to formulate compositions that release prefume at desired stages of the laundry process. For example, different perfumes may be released during storage of the composition, during its use, and during wearing of fabrics, all by selecting silica particles providing different degrees of perfume protection in the presence of peroxyacid bleach.

DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to granular laundry detergent or bleaching compositions containing peroxyacid bleaches and perfumed silica particles.

The Perfume Particles

Silica particles are used as carriers for perfumes to make dry flowable perfume compositions. In general, it is desired that the total amount of perfume to achieve the desired impact level on dry fabric be adsorbed on the silica. The perfume oil adsorption is affected by particle size (microns) and surface area (m^2/g). In general, the amount of perfume that can be adsorbed per unit weight of silica is greater for small particle sizes. However, it is usually preferred not to load the perfume particles to the maximum loading. Perfume to silica particle weight ratios can range from about 0.001:1 to about 6:1, depending upon the silica particle, with the preferred ratios being from about 0.1:1 to about 3:1, more preferably from about 0.2:1 to about 2.5:1.

The perfume can be sprayed or otherwise loaded onto the silica in various ways well known in the trade.

The perfume compositions of this invention are conventional compositions known in the art which are not also considered to be flavors. Selection of any perfume or amount of perfume is based solely on aesthetic considerations. Suitable perfume compositions can be found in the art including U.S. Pat. Nos. 4,145,184, Brain and Cummins, issued Aug. 26, 1980; 4,209,417, Whyte, issued Jan. 24, 1980; 4,515,705, Moeddel, issued May 7, 1985; and 4,152,272, Young, issued May 1, 1978; all of said patents being incorporated herein by reference. Desirably, the perfume compositions are relatively substantive to maximize the effect on fabrics, although nonsubstantive perfumes can also be used.

The perfumes are adsorbed onto silica particles, preferably fumed silica particles. The silica particles have a particle size of from about 0.001 micron to about 15 microns, preferably from about 0.007 micron to about 5 microns, most preferably from about 0.007 to about 2.5 microns, and even more preferably from about 0.007 micron to about 0.25 micron. The surface area is from about 100 to about 800 m^2/g, preferably from about 200 to about 400 m^2/g. It is desirable to use a larger amount of silica particles than the minimum amount necessary to adsorb the perfume composition. Use of lower ratios of perfume to silica provides improved protection of the perfume. In finished detergent or bleaching products, the silica particles are used at a level of from about 0.001% to about 5%, preferably from about 0.001% to about 2%, more preferably from about 0.1% to about 1%, to provide a level of perfume of from about 0.001% to about 1.5%, preferably from about 0.01% to about 0.5%, all by weight. These very small particle size silicas should be added in a way to minimize dusting, e.g., with an agglomerating aid and/or dust suppressor. The dust suppressor should not be water since that will release the perfume prematurely.

Silica gel particles include Syloid® Silicas such as Numbers: 72; 74; 221; 234; 235; 244; etc. Syloid® silicas are available from W. R. Grace & Co., Davison Chemical Division, P.O. Box 2117, Baltimore, Maryland 21203. Such particles have surface areas of from about 250 to about 340 m^2/g; pore volumes of from about 1.1 to about 1.7 cc/g; and average particle sizes of from about 1 to about 8 microns, preferably from about 2.5 to about 6 microns. Fumed silica particles have primary particle diameters of from about 0.007 to about 0.025 micron and include Cab-O-Sil® Numbers L-90; LM-130; LM-5; M-5; PTG; MS-55; HS-5; and EH-5. Cab-O-Sil® silicas are available from Cabot Corp, P.O. Box 188, Tuscala, Illinois 61953.

In a preferred aspect of this invention, the perfume silica particles can be used to release perfume when they are wetted, e.g., with an aqueous fluid. When the particles contact substrates such as fabrics, skin, absorbent materials, etc., they can be activated upon wetting. When the aqueous material is undesirable such as sweat, urine, menses, etc., the perfume can be either a masking aid or an aesthetically pleasing "signal" that other action is required.

The Peroxyacid Bleach

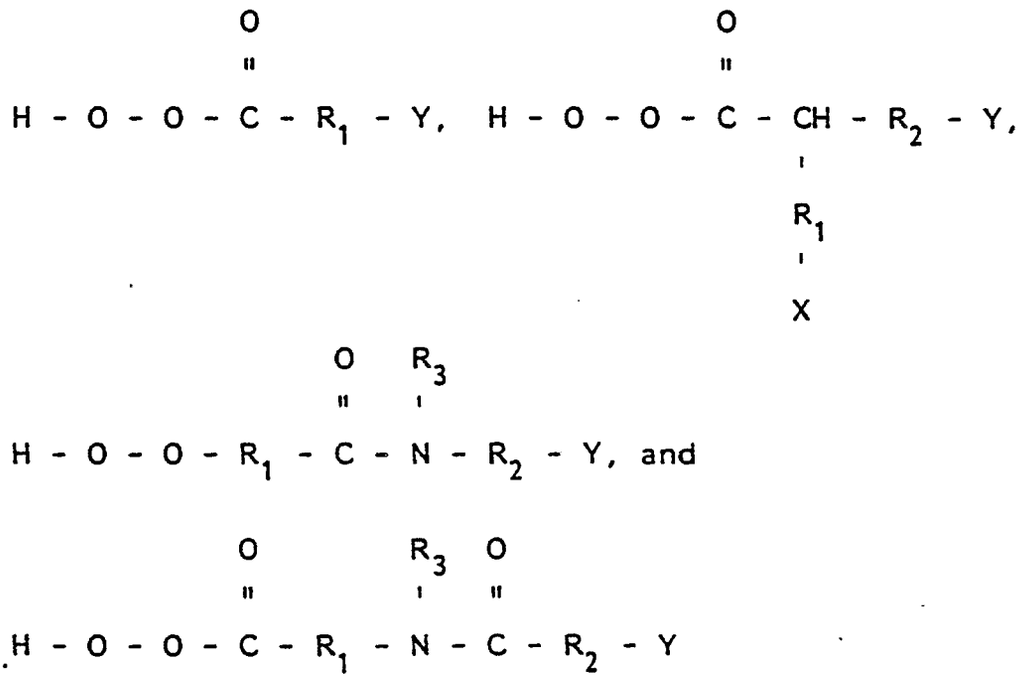
The peroxyacid can be a preformed peroxyacid, an inorganic persalt (e.g., sodium perborate), or a combination of an inorganic persalt and an organic peroxyacid precursor which is converted to a peroxyacid when the combination of persalt and precursor is dissolved in water. The organic peroxyacid precursors are often referred to in the art as bleach activators.

Preferably, the peroxyacid is a preformed peroxyacid. These bleaches are much more reactive with and destructive of perfume ingredients, particularly when in intimate mixture or contact, so that the perfume stability and delivery benefits provided by the present invention are greater than in the case where the bleach is an inorganic persalt or a persalt plus activator.

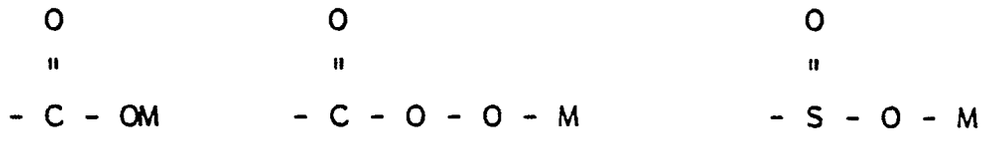
Examples of suitable organic peroxyacids are disclosed in U.S. Patents 4,374,035, Bossu, issued Feb.

15, 1983; 4,681 ,592, Hardy et al, issued July 21, 1987; 4,634,551, Burns et al, issued Jan. 6, 1987; 4,686,063, Burns, issued Aug. 11, 1987; 4,606,838, Burns, issued Aug. 19, 1986; and 4,671,891, Hartman, issued June 9, 1987. Examples of compositions suitable for laundry bleaching which contain perborate bleaches and activators therefor are disclosed in U.S. Patents 4,412,934, Chung and Spadini, issued Nov. 1, 1983; 4,536,314, Hardy et al, issued Aug. 20, 1985; 4,681,695, Divo, issued July 21, 1987; and 4,539,130, Thompson et al, issued Sept. 3, 1985. All of the above patents are incorporated herein by reference.

The preferred organic peroxyacid is selected from the following:



wherein R_1 and R_2 are alkylene groups containing from 1 to about 20 carbon atoms or phenylene groups, R_3 is hydrogen or an alkyl, aryl, or alkaryl group containing from about 1 to about 10 carbon atoms, and X and Y are hydrogen, halogen, alkyl (e.g., methyl, isopropyl), aryl, or any group which provides an anionic moiety in aqueous solution. Such X and Y groups can include, for example,



where M is hydrogen or a water-soluble salt-forming cation. Mixtures of such peroxyacids can also be used herein.

Specific examples of preferred peroxyacids for this invention include diperoxydodecanedioic acid (DPDA), nonylamide of peroxysuccinic acid (NAPSA), and decyldiperoxysuccinic acid (DDPSA). For the purpose of this invention, the peroxyacid is preferably incorporated into a soluble granule according to the method described in the above cited U.S. Pat. No. 4,374,035. A preferred bleach granule comprises, by weight, 1% to 50% of an exotherm control agent (e.g., boric acid); 1% to 25% of a peroxyacid compatible surfactant (e.g., C_{13}LAS); 0.1% to 10% of one or more chelant stabilizers (e.g., sodium pyrophosphates); and 10% to 70% of a water-soluble processing salt (e.g., Na_2SO_4).

The peroxyacid bleach is used at a level which provides an amount of available oxygen (AvO) from about 0. 1% to about 10%, preferably from about 0.5% to about 5%, and most preferably from about 1% to about 4%, all by weight of the composition.

Effective amounts of peroxyacid bleach per unit dose of the composition of this invention used in typical laundry liquor, e.g., containing 64 liters of 16°-60° C water, provide from about 1 ppm to about 150 ppm of available oxygen (AvO), more preferably from about 5 ppm to about 50 ppm. The laundry liquor should also have a pH of from 7 to 10, preferably 7.5 to 9, for effective peroxyacid bleaching. See Col. 6,

lines 1-10, of U.S. Pat. No. 4,374,035.

Alternatively, the composition may contain a suitable organic precursor which generates one of the above peroxyacids when reacted with alkaline hydrogen peroxide in aqueous solution. The source of hydrogen peroxide can be any inorganic peroxygen compound which dissolves in aqueous solution to generate hydrogen peroxide, e.g., sodium perborate (monohydrate and tetrahydrate) and sodium percarbonate.

These compositions comprise:

(a) a peroxygen bleaching compound capable of yielding hydrogen peroxide in an aqueous solution; and

(b) a bleach activator having the general formula:

wherein R is an alkyl group containing from about 5 to about 18 carbon atoms wherein the longest linear alkyl chain extending from and including the carbonyl carbon contains from about 6 to about 10 carbon atoms and L is a leaving group, the conjugate acid of which has a pK_a in the range of from about 6 to about 13.

Detergent or Bleaching Compositions

The perfume particles of the present invention are preferably formulated into laundry detergent or bleaching compositions. In a preferred embodiment, the bleaching compositions of the invention are also detergent compositions. Such compositions typically comprise detersive surfactants and/or detergency builders and, optionally, additional ingredients such as enzymes, fabric brighteners and the like. The perfume particles are present in the detergent composition at a level of from about 0.001% to about 2%, and preferably from about 0.1% to about 1%, by weight of the detergent composition. The remainder of the detergent composition will comprise from about 1% to about 50%, preferably from about 10% to about 25%, detersive surfactant, and from about 10% to about 80%, preferably from about 20% to about 50%, of a detergency builder, and, if desired, other optional laundry detergent components.

1. The Surfactant

Surfactants useful in the detergent compositions herein include well-known synthetic anionic, nonionic, amphoteric and zwitterionic surfactants. Typical of these are the alkyl benzene sulfonates, alkyl- and alkylether sulfates, paraffin sulfonates, olefin sulfonates, alkoxylated (especially ethoxylated) alcohols and alkyl phenols, amine oxides, alpha-sulfonates of fatty acids and of fatty acid esters, alkyl betaines, and the like, which are well known from the detergency art. In general, such detersive surfactants contain an alkyl group in the C_9-C_{18} range. The anionic detersive surfactants can be used in the form of their sodium, potassium or triethanolammonium salts; the nonionics generally contain from about 5 to about 17 ethylene oxide groups. C_{11}-C_{16} alkyl benzene sulfonates, C_{12}-C_{18} paraffin-sulfonates and alkyl sulfates are especially preferred in the compositions of the present type.

A detailed listing of suitable surfactants for the detergent compositions herein can be found in U.S. Pat. No. 3,936,537, Baskerville, issued Feb. 3, 1976, incorporated by reference herein. Commercial sources of such surfactants can be found in McCutcheon's EMULSIFIERS AND DETERGENTS, North American Edition, 1984, McCutcheon Division, MC Publishing Company, also incorporated herein be reference.

2. Detergency Builders

Useful detergency builders for the detergent compositions herein include any of the conventional inorganic and organic water-soluble builder salts, as well as various water-insoluble and so-called "seeded" builders.

Nonlimiting examples of suitable water-soluble, inorganic alkaline detergent builder salts include the alkali metal carbonates, borates, phosphates, polyphosphates, tripolyphosphates, bicarbonates, silicates, and sulfates. Specific examples of such salts include the sodium and potassium tetraborates, bicarbonates,

carbonates, tripolyphosphates, pyrophosphates, and hexametaphosphates.

Examples of suitable organic alkaline detergency builder salts are: (1) water-soluble amino polyacetates, e.g., sodium and potassium ethylenediaminetetraacetates, nitrilotriacetates, and N-(2-hydroxyethyl)-nitrilodiacetates; (2) water-soluble salts of phytic acid, e.g., sodium and potassium phytates; (3) water-soluble polyphosphonates, including sodium, potassium and lithium salts of ethane-1-hydroxy-1,1-diphosphonic acid, sodium, potassium, and lithium salts of methylenediphosphonic acid and the like.

Seeded builders include such materials as sodium carbonate or sodium silicate, seeded with calcium carbonate or barium sulfate. Hydrated sodium Zeolite A having a particle size of less than about 5 microns is particularly desirable.

A detailed listing of suitable detergency builders can be found in U.S. Pat. No. 3,936,537, incorporated herein by reference.

3. Optional Detergent Ingredients

Optional detergent composition components include enzymes (e.g., proteases and amylases), halogen bleaches (e.g., sodium and potassium dichloroisocyanurates), soil release agents (e.g., methylcellulose), soil suspending agents (e.g., sodium carboxymethylcellulose), fabric brighteners, enzyme stabilizing agents, color speckles, suds boosters or suds suppressors, anticorrosion agents, dyes, fillers, germicides, pH adjusting agents, nonbuilder alkalinity sources, and the like.

The following nonlimiting examples illustrate the compositions of the present invention.

All percentages, parts, and ratios used herein are by weight unless otherwise specified.

EXAMPLE I

The formulation hereinafter described is a perfumed silica gel made on a lab scale according to the following method.

A predetermined amount of silica gel is placed into a Cuisinart® food processor and a fluid bed state is achieved by the action of the processor's blades. Knowing the desired amount of perfume impact on dry fabric and, hence, the desired perfume to silica gel ratio, the premeasured perfume is added through a small orifice into the fluid bed of silica gel until all the perfume has been applied. Mixing is continued until the perfume and silica gel have reached a homogenous dry flowable state.

Perfume/silica gel composition is prepared as follows:

Ingredient	% by Weight
Syloid® 234*	51.61
Perfume	48.39
Total	100.00

*Available from W. R. Grace & Co., Davison Chemical Division, P.O. Box 2117, Baltimore, Maryland 21203. Average particle size 2.5 microns on a weight basis and surface area of 250 m^2/g.

Two different perfumes are as follows:

Substantive Perfume (A)		Relatively Nonsubstantive Perfume (B)	
Component	Wt.%	Component	Wt.%
Benzyl Acetate	5.0	Linalool	5.0
Benzyl Salicylate	10.0	Cedarwood Terpenes	20.0
Coumarin	5.0	Dihydro Myrecenol	10.0
Ethyl Maltol	5.0	Eugenol	5.0
Ethylene Brassylate	10.0	Lavandin	15.0
Galoxolide® (50%)	15.0	Ionone Alpha	5.0
Hexyl Cinnamic Aldehyde	20.0	Phenyl Ethyl Acetate	5.0
Inone Gamma Methyl	10.0	Citronellol	10.0
Lilial®	15.0	Geraniol	5.0
Patchouli	5.0	Phenyl Ethyl Alcohol	20.0
Total	100.0	Total	100.0

Other perfumed silica particles of the present invention are obtained when the above perfumes are added with an eye dropper to beakers containing Cab-O-Sil® EH-5 particles (average particle size of 0.007 microns and surface area of about 380 m^2/g, available from Cabot Corporation, Cab-O-Sil Division, P. O. Box 188, Tuscola, Illinois 61953) in a weight ratio of perfume to silica particle of about 2:1, 2.5:1 and 3:1. After shaking the particles for about 1 minute, they become free flowing.

EXAMPLE II

Perfumed granular detergent and bleaching compositions are prepared by mixing the following ingredients.

Ingredient	Parts
1. Spray-dried base granules	
Sodium C_{12} linear alkyl benzene sulfonate	9.14
Sodium C_{14}-C_{15} fatty alcohol sulfate	3.92
Ethoxylated (6 avg.) C_{12}-C_{13} fatty alcohol	0.44
Sodium sulfate	31.60
Sodium silicate (2.0r)	2.40
Sodium polyacrylate (M.W. 4000)	0.98
Sodium tripolyphosphate	4.63
Sodium diethylenetriamine pentaacetate	0.40
Sodium carbonate	13.00
Optical brightener	0.36
Moisture	3.50
Total	70.41
2. Admix	
Sodium acid pyrophosphate	14.51
Peracid bleach*	10.66
Protease enzyme (Alcalase)	0.55
Perfume particles**	0.45
Silicone suds suppressor	0.28
Sodium perborate monohydrate	0.60
Sodium sulfate	2.54
Total	100.00

* Dry granular particles containing 24.00% nonylamide of peroxysuccinic acid, 3.27% nonylamide of succinic acid, 12.00% boric acid, 5.21% C_{13} linear alkylbenzene sulfonic acid, 55.29% sodium sulfate, 0.12% dipicolinic acid, 0.05% phosphoric acid, and 0.06% trisodium phosphate, prepared as described in U.S. Patents 4,374,035, Bossu, and 4,686,063, Burns, both incorporated herein by reference.

** Perfumed particles of Example I. A preferred composition contains a weight ratio of perfume to the Cab-O-Sil^{R} EH-5 particles of about 2:1.

The silica particles protect the perfume from the peroxyacid bleach so that the perfume can be carried through the laundry washing and drying stages and delivered to the fabric.

EXAMPLE III

Perfumed particles are prepared by first mixing Syloid® 234 with the perfumes of Example I to form the following perfume particle compositions according to a process similar to that of Example I:

Perfumed Syloid®	
Ingredient	% by Weight
Syloid® 234	70.6
Perfume	29.4
Total	100.00

The Syloid and the perfume are blended by first adding 30 lbs. of the Syloid® 234 to a Littleford Model FM 130 D Mixer (Littleford Bros., Inc., 15 Empire Drive, Florence, Kentucky 41042). With the plow turned on, the perfume is slowly introduced dropwise through a 3/8" pipe at a rate of approximately 2-2.5 lbs/min. After 12.5 lbs. of perfume are added, the chopper is turned on for 15 seconds to evenly disperse the perfume before emptying the mixer.

These perfumed particles are used in the composition of Example II.

EXAMPLE IV

Other granular detergent and bleaching composition herein are prepared by mixing the following ingredients with the perfumed particles of Examples I and III.

Ingredient	Parts
1. Spray-dried base granules	
Sodium C_{13} linear alkyl benzene sulfonate	9.76
Sodium C_{14}-C_{15} fatty alcohol sulfate	4.18
Ethoxylate (6 avg.) C_{12}-C_{13} fatty alchol	1.33
Sodium sulfate	13.15
Sodium silicate (1.6r)	4.21
Sodium polyacrylate (M.W. 4,000)	0.65
Sodium tripolyphosphate	21.49
Sodium pyrophosphate	5.22
Sodium diethylenetriamine pentaacetate	0.38
Sodium carbonate	22.03
Polyethylene glycol (M.W. 8000)	0.43
Optical brightener	0.18
Moisture	6.52
Total	70.41
2. Admix	
Protease enzyme (Savinase)	0.56
Perfume particles	0.45
Sodium perborate monohydrate	4.04
Bleach activator*	5.58

* Cylindrical particles, 0.9 mm dia. x 1.5 - 3.0 mm long, containing 83% sodium linear nonyloxybenezene sulfonate, 5% polyethylene glycol 8000, 5% palmitic acid and 3% sodium C_{13} linear alkylbenzene sulfonate.

Other compositions of the present invention are obtained when the phosphate builders in the composi-

tions of Examples II and IV are replaced with hydrated Zeolite A (avg. diameter of 2 microns) or with an 80:20 by weight mixture of the tartrate monosuccinate:tartrate disuccinate builders described in U.S. Patent 4,663.071, Bush et al, issued May 5, 1987.

Claims

1. A granular laundry detergent or bleaching composition comprising a peroxyacid bleach, characterized in that it further comprises a dry, flowable, perfumed silica particle, preferably from 0.001% to 5% of the silica particle, having a particle size of from 0.001 micron to 15 microns and having a suitable perfume composition adsorbed onto said silica particle, the weight ratio of said perfume composition to the silica particle being from 0.001:1 to 6:1, preferably from 0.1:1 to 3:1.

2. The composition of Claim 1 wherein said silica particle is a silica gel having a particle size of from 1 micron to 8 microns.

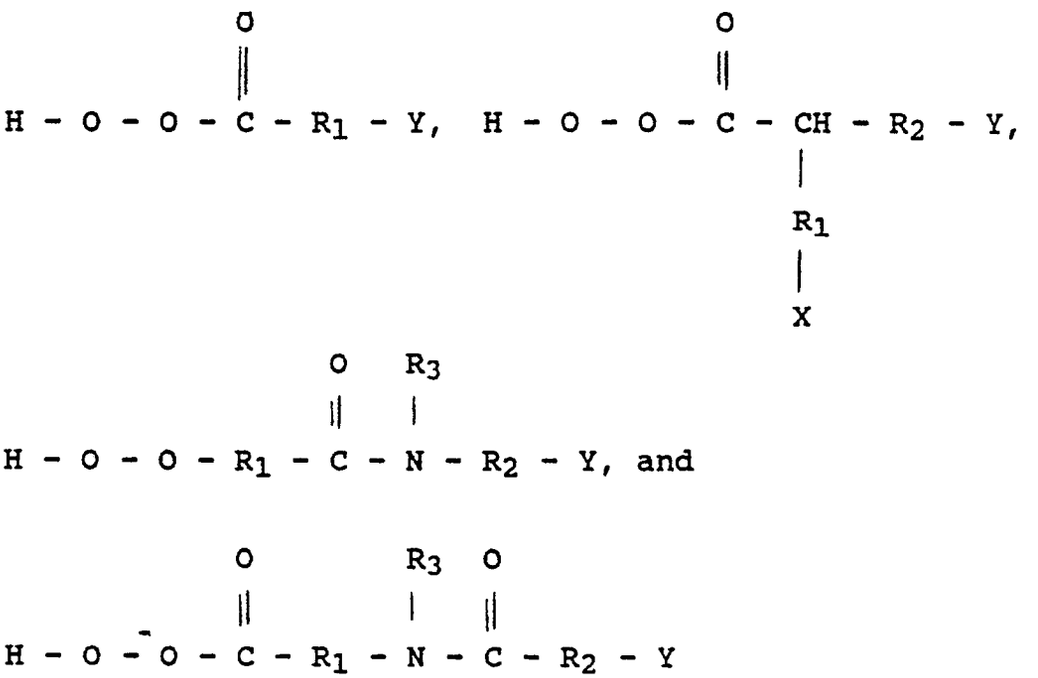
3. The composition of Claim 1 wherein said silica particle is a fumed silica having a particle size of from 0.007 microns to 0.025 microns and a surface area of from 200 to 400 m^2/g.

4. The composition of any one of the preceding claims wherein said silica particle is present at a level of from 0.1% to 1% to provide a perfume level of from 0.01% to 0.5%.

5. The composition of any one of the preceding claims wherein said perfume composition is relatively substantive to said fabrics.

6. The composition of any one of the preceding claims wherein the peroxyacid bleach is a preformed peracid present at a level to provide from 0.1% to 10% of available oxygen.

7. The composition of any one of the preceding claims wherein the peracid is selected from the group consisting of:



wherein R_1 and R_2 are alkylene groups containing from 1 to 20 carbon atoms or phenylene groups, R_3 is hydrogen or an alkyl, aryl, or alkaryl group containing from 1 to 10 carbon atoms, and X and Y are hydrogen, halogen, alkyl, aryl, or any group which provides an anionic moiety in aqueous solution.

8. The composition of any one of the preceding claims wherein the peracid is diperoxydodecanedioic acid or the nonylamide of peroxysuccinic acid.