

1

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TEXTILE BLEACHING COMPOSITION

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This invention relates to compositions of matter which are useful for the bleaching of textiles and other materials, particularly those made of the synthetic linear polyamides known as nylon.

The bleaching of nylon materials, especially textile materials which are to be used in the white or undyed state, has presented a problem which has not been heretofore satisfactorily solved. Undyed nylon fabrics which have been used for some time may become slightly yellow or otherwise discolored. The use of alkaline peroxide bleach solutions results in excessive damage to nylon fabrics, while other bleaching compositions which have been proposed are either not sufficiently effective or are very inconvenient to use. The use of hydrogen peroxide in mildly acidic solutions, as described in U.S. 2,720,441, requires an excessive length of time for bleaching, while its use in strongly acidic solutions leads to excessive strength losses. The most effective bleach available heretofore has included alkali metal hypochlorites and acetic acid. This method of bleaching, however, is inconvenient and the results are dependent on accurate measurement of the ingredients in home laundering. Thus, the lack of a convenient and effective home bleach for nylon reduces the potential of nylon in several of the larger markets for white goods and also in the full utilization of nylon in many of its present end uses, such as end blends with cotton.

It is accordingly an object of this invention to provide an improved bleaching composition. Another object is to provide a composition of matter which may be conveniently and effectively used for home bleaching of nylon textile materials. A further object is to provide a bleaching composition which may be used in conjunction with soaps and detergents which are commonly used in home laundering of nylon and other textiles.

The above objects are accomplished according to this invention by a bleaching composition comprising a solid, stable peroxygen compound and an N-acylimide having the general formula:



wherein R is aromatic or substituted aromatic and R₁ is an imide radical, preferably derived from a cyclic imide.

The above compositions are superior to any presently available home bleach for nylon in that they are highly effective in removing discoloration from nylon fabrics without causing excessive strength losses, and in that they may be conveniently and effectively used alone or in combination with household soaps or detergents for bleaching purposes.

Although it has been customary to judge fabric color by eye, it is much more satisfactory to use precise instrumental methods whereby a numerical value can be obtained. In the following examples, color measurements are made using a differential colorimeter as described by L. B. Glasser and D. J. Troy in the Journal of the Optical Society 42, 652-660 (1952). These authors describe a method whereby reflectance measurements can be converted into the color coordinates, L for lightness, *a* for greenness-redness, and *b* for yellowness-blueness. Using this system, negative (−) values of *b* represent blue shades, while positive (+) values represent yellow shades. The depth or intensity of color will be propor-

2

tional to the numerical magnitude of the *b* value. Also described is a method for determining the total color difference (ΔE) between a sample and a white standard when the sample contains an admixture of colors which cannot be described in terms of any single color coordinate.

In the following examples, bleaching tests are carried out on fabrics which have been discolored by either dye transfer or heat yellowing, since these are the most common sources of garment discoloration encountered in actual use. The dye transferred fabrics are discolored by laundering white fabrics in the presence of blue, red, and yellow fabrics which had been dyed to saturation with certain dyes of poor washfastness. The fabrics used for this purpose in the following examples are dyed with a blue dye, Anthraquinone Blue SWF (Prototype No. 12, Technical Manual and Yearbook, A.A.T.C.C., 1955), a red dye, Celanthrene Red 3BN (Prototype No. 234), and a yellow dye, Celanthrene Fast Yellow GL (Prototype No. 534). The color of the dye transferred fabrics is numerically described in the examples in terms of the total color difference (ΔE) when compared to the same fabrics before discoloration. Thus, the lower the total color difference (ΔE), after treatment with an oxidizing agent, the greater the color removal and the more effective the bleach. The heat-yellowed fabrics described in the following examples are discolored by exposure to excessive heat. The color of these fabrics is described in terms of the +*b* value (yellowness). The effectiveness of the bleaching action of the various compounds given in the examples can be clearly determined by comparison of the magnitude of the +*b* values of a fabric, before and after bleaching.

EXAMPLE I

Nylon taffeta fabrics, discolored by exposure to heat and by laundering in the presence of dyed fabrics, are immersed in an aqueous bath buffered to pH 6 with citric acid-disodium phosphate, the weight of the bath being 50 times the weight of the fabric. A mixture of N-benzoyl succinimide and anhydrous sodium perborate is added to the bath and the fabrics allowed to soak for one hour at a temperature of 45° C. The fabric samples are then removed from the bath, rinsed and dried, and the total color (delta E) of the samples before and after bleaching is determined with a differential colorimeter as described above. Table I below shows the results obtained with various molar ratios of sodium perborate to N-benzoyl succinimide and with various concentrations of the bleaching composition. The concentration of bleaching composition is expressed as percent based on the weight of the fabric sample. Total color measurements before and after bleaching are shown for each of the samples. Also shown, for comparison, is the result obtained when sodium perborate is used alone under these conditions.

Measurement of the Mullen bursting strength of the fabric before and after bleaching showed a strength loss of less than 2.0% in all cases.

Table I

Molar Ratio	Concentration, Percent	ΔE	
		Before	After
Sodium perborate alone.....	0.2	9.4	9.1
2.5.....	0.4	9.4	1.3
2.5.....	0.6	9.4	1.5
6.0.....	0.34	8.8	1.0
6.2.....	0.7	9.4	1.2
12.4.....	0.6	9.4	1.2

When the buffering agents, citric acid and disodium

3

phosphate, are mixed with the dry bleaching composition and the weight of bleaching composition increased sufficiently to provide the same concentration of N-benzoyl succinimide and sodium perborate in the bleaching bath, similar results are obtained.

EXAMPLE II

Samples of nylon taffeta fabric are treated as described in Example I except that various N-substituted imides were substituted for N-benzoyl succinimide. Table II below shows the compounds used, the molar ratio of anhydrous sodium perborate to imide and the total color before and after bleaching. A sufficient amount of the bleach composition is added to give a concentration in each case of 0.34%, based on the weight of the fabric sample.

Table II

	Molar Ratio	ΔE	
		Before	After
N-p-anisoylsuccinimide.....	7.0	14.5	5.4
N-alpha-naphthoylsuccinimide.....	7.5	9.4	3.2
N-beta-naphthoylsuccinimide.....	7.5	9.4	2.4
N-benzoylglutarimide.....	6.4	8.5	4.1
N-p-chlorobenzoylsuccinimide.....	7.0	14.5	7.2

EXAMPLE III

Samples of nylon taffeta fabric are bleached for one hour at 45° C. in an aqueous bath to which has been added a mixture of N-benzoyl 5,5-dimethylhydantoin and sodium perborate, the total weight of the bleaching bath being 100 times the weight of the sample. The bleach bath is maintained at a pH of 6.7 by the addition of citric acid and disodiumphosphate. Table III shows the reduction in color obtained with various concentrations of N-benzoyl 5,5-dimethylhydantoin based on the weight of the fabric and various molar ratios of sodium perborate to N-benzoyl dimethylhydantoin.

Table III

Concentration Percent	Molar Ratio	ΔE	
		Before	After
0.1.....	7.2	8.5	1.7
0.2.....	3.6	8.5	1.1
0.3.....	2.4	8.5	1.2
0.5.....	1.4	8.5	1.0

EXAMPLE IV

Example I is repeated except that potassium monopersulfate, KHSO_5 , is substituted for sodium perborate. Table IV below shows the molar ratio of potassium monopersulfate to N-benzoyl succinimide in the bleaching composition, the concentration of the bleaching composition based on the weight of the fabric and the total color before and after bleaching.

Table IV

Molar Ratio	Concentration, Percent	ΔE	
		Before	After
5.75.....	0.53	9.4	2.5

EXAMPLE V

A bleaching composition is prepared by mixing 24.9% by weight of N-benzoyl succinimide, 39.0% by weight of anhydrous sodium perborate, 33.3% by weight of citric acid, 2.54% by weight of an anionic detergent (sodium

4

lauryl sulfate) and 0.26% by weight of a commercial fluorescent whitening agent. Nylon taffeta fabric samples are bleached with this composition, using the conditions given in Example I except that sufficient bleach is added to the bath to give a concentration of 1.0%, based on the weight of the fabric sample. The total color of the samples is determined before and after bleaching as described in Example I. Table V below shows results obtained with the freshly prepared bleaching composition and after storage for six and ten weeks in both glass containers and cardboard cartons. As shown in the table, no substantial loss in bleaching power is observed.

Table V

	ΔE	
	Before	After
Fresh bleaching composition.....	13.8	1.8
After 6 weeks' storage in glass.....	13.8	3.5
After 10 weeks' storage in glass.....	13.8	3.3
After 6 weeks' storage in cardboard.....	13.8	3.3
After 10 weeks' storage in cardboard.....	13.8	3.3

EXAMPLE VI

This example illustrates the effectiveness of the bleaching compositions of this invention at various concentrations and various molar ratios of peroxygen compound to N-substituted imide. Samples of nylon taffeta fabric are bleached for one hour at 45° C. in an aqueous bleaching bath containing N-benzoyl succinimide and sodium perborate, the weight of bath being 50 times the weight of the sample. The concentration of N-benzoyl succinimide is kept constant at 0.2%, based on the weight of the fabric sample, while the molar ratio of sodium perborate to N-benzoyl succinimide is varied as shown in Table VI below. The pH of the bleach bath is varied by changing the relative concentration of the buffers. For pH 6 and 7, a citric acid-disodium phosphate buffer is used, while for pH 8 and 9, a boric acid-sodium hydroxide buffer is employed to give the values shown below. The reduction in total color and yellowness (+b) at various pH levels and various molar ratios is shown in Table VI-A below. Table VI-B shows the reduction in total color and yellowness obtained with various concentrations of N-benzoyl succinimide and various molar ratios of sodium perborate to N-benzoyl succinimide when the aqueous bleaching bath is maintained at a pH of 10.

Table VI-A

pH	Molar Ratio	ΔE	Yellowness
Control.....		18.4	7.2
6.....	1.0	6.5	4.6
6.....	2.0	6.1	3.9
6.....	3.0	5.2	3.4
6.....	6.0	5.4	3.3
7.....	0.6	6.1	4.3
7.....	1.0	5.5	4.0
7.....	2.0	4.3	3.6
7.....	3.0	4.1	3.2
8.....	6.0	5.1	3.4
8.....	0.6	5.5	4.1
8.....	1.0	4.7	3.4
8.....	2.0	4.5	3.3
8.....	3.0	5.3	3.7
9.....	6.0	10.4	3.8
9.....	0.6	5.4	3.7
9.....	1.0	5.2	3.5
9.....	2.0	8.2	3.6
9.....	3.0	13.4	4.2
9.....	6.0	16.8	5.3

5
Table VI-B

Concentration of N-benzoyl succinimide, percent	Molar Ratio	Total Color	Yellowness
Control		8.8	7.0
0.2	0.1	4.2	5.5
0.2	0.3	4.2	5.4
0.2	0.6	3.7	4.4
0.2	0.8	3.4	4.2
0.2	1.0	4.1	4.5
0.2	1.2	5.7	5.1
0.15	0.6	3.9	4.5
0.15	0.8	3.8	4.6
0.15	1.0	4.1	5.3
0.10	0.6	4.3	5.2
0.10	0.8	4.5	5.2
0.10	1.0	4.5	5.3

EXAMPLE VII

A bleaching composition is prepared by mixing 42% by weight of N-benzoyl succinimide, 12.5% by weight of sodium perborate, 42.5% by weight of sodium carbonate, 2.6% by weight of a detergent (sodium lauryl sulfate) and 0.4% of a fluorescent whitener (Tinopal RBS). Swatches (6 x 6 inches) cut from a heat-yellowed commercial cotton percale sheet are bleached in a solution to which has been added 1% (based on fabric weight) of the above bleach composition for one hour at 45° C. Colorimeter analysis showed the original fabric to have a yellowness value of 0.3 unit, which is increased to 3.5 units after exposure to heat. After bleaching, the fabrics have a yellowness value of 0.4 unit. Measurement of the fluidity of the sample, which is a measure of the extent of degradation due to bleaching, using test method 82-54 of the Technical Manual and Year Book of the American Association of Textile Chemists and Colorists, showed no appreciable change after three bleaching cycles. When the same fabric was bleached with alkali-chlorite and vinegar, the fluidity after one bleaching increased markedly and after three bleachings was up to a value of 20.2, as compared to 3.7 for the bleaching composition of this invention.

EXAMPLE VIII

Example VII is repeated using the bleaching composition of Example V with substantially the same results.

A wide range of pH values can be used in bleaching fabrics with the compositions of this invention without appreciable damage to the fabrics. The desired pH may be achieved by adding suitable well-known buffering agents to the bleaching solution or by mixing the buffering agents with the bleaching composition in the dry state, if desired. The bleaching effectiveness of these compositions at high pH values permits their use in combination with common household laundry soaps and detergents for preventive bleaching of nylon and other fibers. Likewise, almost any of the commercially available optical whiteners may be used in combination with these compositions. At a pH in the range of about 6 to about 10, the bleach is most effective and fiber degradation is minimized considerably.

Preferably, the bleaching composition comprises N-benzoyl succinimide and sodium perborate, either anhydrous or hydrated, since this composition is highly effective and the ingredients are readily available and relatively inexpensive.

Suitable N-acylimides, in addition to those given in the examples, include: N,N-dibenzoylformamide, N,N-dibenzoyl acetamide, 1,3-dibenzoyl dimethylhydantoin, N-benzoylphthalimide, N-benzoylpyrrolidone, N-cinnamoylsuccinimide, and N - m - nitrobenzoylsuccinimide. Preferably, cyclic N-substituted imides are employed. R in the formula



6

is preferably an aromatic or substituted aromatic radical, aliphatic radicals being much less effective.

Any peroxygen compound which is stable in the dry state and which liberates active oxygen readily when dissolved in aqueous media may be employed. Suitable peroxygen compounds, in addition to those given in the examples, include alkali percarbonates such as sodium and potassium percarbonate, urea peroxide, zinc peroxide, calcium peroxide, magnesium peroxide, tetrasodium pyrophosphate, and disodium perphosphate and others.

The molar ratio of peroxygen compound to N-acylimide may vary widely depending on the conditions employed. As little as 0.1 mol of peroxygen compound per mol of N-acylimide is effective, while as much as 12 mols of peroxygen compound per mol of N-acylimide is not harmful at the lower pH levels. However, when the bleach composition is used at high pH values, e.g., pH 10, the molar ratio is preferably no higher than about 2 for optimum bleaching.

The bleaching compositions of this invention are particularly suitable for bleaching nylon textiles but may also be used for bleaching cotton, regenerated cellulose, and textile materials made from various synthetic fibers such as polyesters, polyacrylonitrile and polyurethane fibers.

The concentration of the bleaching composition in an aqueous solution, as well as the time and temperature involved, may be adjusted according to the particular use anticipated. For example, as a preventive bleach to be used during laundering of nylon fabrics to prevent the build-up of discoloration, a concentration between about 0.02 and about 0.2%, based on the weight of solution, is satisfactory. For the latter use, an average washing cycle (10-20 minutes) of a home automatic washing machine using water at a temperature of about 30° to about 60° C., is satisfactory. As a corrective bleach to whiten badly discolored fabrics, a concentration of about 0.1 to about 2.0%, based on the weight of solution, with a washing cycle of about 20 to about 60 minutes with water at about 30° to about 100° C., is satisfactory.

I claim:

1. A textile bleaching composition consisting essentially of a solid, stable peroxygen compound and a water soluble N-acylimide having the general formula:



wherein R comprises an aromatic group and R₁ comprises an imide radical, the molar ratio of the peroxygen compound to imide being in the range of from about 0.1 to about 12.4.

2. The bleaching composition of claim 1 wherein the peroxygen compound is sodium perborate.

3. The bleaching composition of claim 1 wherein the N-acylimide is N-benzoyl succinimide.

4. A textile bleaching composition consisting essentially of sodium perborate and N-benzoyl succinimide in a molar ratio of from about 0.1 to about 12.4.

5. The bleaching composition of claim 4 in aqueous solution, said solution containing at least about 0.1% by weight thereof.

6. The bleaching solution of claim 5 buffered to a pH of 6-10.

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