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PEROXYGEN BLEACHING OF NYLON

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This invention relates to a method for bleaching nylon and particularly nylon textile materials.

"Nylon" is a generic term used to designate synthetic linear polyamides obtainable from bifunctional reactants as described in U. S. Patents 2,071,253 and 2,130,940. Polyhexamethylene adipamide is typical of such polyamides. Nylon textile materials e. g. fibers, yarns and fabrics, etc. are widely known.

The bleaching of nylon materials has presented somewhat of a problem. The usual alkaline peroxide bleach solutions damage nylon excessively whereas the brightening action of dilute aqueous solutions of peracids such as peracetic acid is not sufficient to make their use practical.

One object of the present invention is to provide an improved method for bleaching nylon materials, particularly nylon textile materials. A further object is to provide an improved method for bleaching such materials with peroxygen compounds whereby excellent brightness is obtained without excessive damage to the nylon material resulting. Still further objects will be apparent from the following description.

The above objects are accomplished in accordance with the invention by subjecting a nylon material, particularly a nylon textile material, to the action of an aqueous bleaching medium having dissolved therein a peroxygen compound and an aliphatic carboxylic acid having a dissociation constant at 25° C. in the range 1.0×10^{-3} to 1.0×10^{-6}.

Bleaching can be effected by contacting the nylon material with the aqueous medium containing the dissolved peroxygen compound and aliphatic carboxylic acid in any desired manner. A simple and effective procedure is to immerse the material in such a medium until the desired bleaching action has occurred. Bleaching occurs effectively but slowly at ordinary temperatures. It is advantageous in order to reduce the bleaching time to operate at somewhat elevated temperatures, e. g., at temperatures in the range 35° C. to the boiling temperature of the medium. A preferred range is 35 to 85° C., the most preferred range being 50 to 70° C. Effective bleaching is usually accomplished in 3 to 5 hours at temperatures of 35 to 85° C. Bleaching is slower at the lower temperatures and more rapid at the higher temperatures. Bleaching can be effected in about ½ hour at the boiling temperature of the medium but operation at such higher temperatures is not generally desirable due to the obnoxious fuming of the bleaching medium.

Pressure is not an important factor insofar as the invention is concerned and any pressure may be used so long as the bleaching medium remains liquid at the chosen temperature of operation.

It is essential that the bleaching medium contain a substantial amount both of water and of dissolved aliphatic carboxylic acid. Media containing about 4 to 70% of dissolved acid and about 30 to 96% of water, based upon the total weight of the medium, are generally satisfactory.

The upper limit for the acid content is not as critical as the lower limit and may vary somewhat from the stated 70%, depending upon the acid used. Generally, however, acid concentrations substantially above 70% will not be used since at such higher concentrations, solution of the nylon material tends to become appreciable and degradation of the nylon increases. On the other hand, bleaching action falls off rapidly as the concentration of the acid is decreased below 4%. The preferred media will contain from about 10 to 50% dissolved acid and from about 90% to 50% of water. Ordinarily, no substantial added advantage results from acid concentrations greater than about 20% to 30%. When using acid concentrations in the preferred range, tensile strength losses seldom exceed 10% and usually are substantially less than 10%.

Illustrative of the aliphatic carboxylic acids which are usable in accordance with the invention are: acetic, hydroxyacetic, monochloroacetic, propionic, α-chloropropionic, β-chloropropionic, citric, formic, isobutyric, lactic, malic, malonic, mandelic, succinic and tartaric acids. The preferred acids are those having a dissociation constant within the range 1.0×10^{-4} to 1.0×10^{-6}. The acid may contain any substituent group or groups on the aliphatic carbon nucleus so long as such group does not react adversely towards the peroxygen constituent of the bleaching medium and the acid is sufficiently soluble. Examples of suitable substituent groups are the halides such as chlorine and bromine and the hydroxyl, cyano, amino and phenyl radicals. The use of formic, acetic or hydroxyacetic acid is generally preferred because of their general suitability and cheapness.

The invention is illustrated by the following examples in which all percentages of reagents represent percentages by weight. All brightness values are reported as percent reflectances and were determined using a Hunter reflectometer in accordance with well-known procedures.

Example 1

About 200 g. of a bleaching medium containing 25% hydroxyacetic acid and 1.0% H_2O_2 was prepared by adding acetic acid and aqueous 35% hydrogen peroxide to water. The pH of the medium was 1.2. Pieces of nylon grey goods (7" x 14") which had previously been scoured and desized with dilute caustic soda solution to a brightness of 71.9% were immersed in the bleaching medium for 4 hours at 65° C. The treated fibers were then washed with water and found to have an average brightness of 83.8%. The average fiber strength loss was 6.2% as determined on a Scott tester following the A. S. T. M. cut strip (⅝" x 6") procedure.

Example 2

About 200 g. of a bleaching medium containing 25% acetic acid and 1.0% H_2O_2 was obtained by adding glacial acetic acid and aqueous 35% hydrogen peroxide to water. Pieces of desized nylon grey goods having a brightness value of 71.9% were immersed in this solution at 65° C. for 4 hours. Following washing with water, the brightness of the treated goods was 84.5%. No strength loss resulted from the treatment.

Example 3

A bleaching medium was prepared as described in Example 2 employing citric acid in place of acetic acid. The brightness of nylon fabric which had been scoured and desized was increased from 72.6% to 81.2% by immersion in this bleaching medium at 65° C. for 4 hours. The strength loss resulting from the treatment was 7%.

Example 4

A 200 g. bleaching medium containing 25% H_2SO_4

and 1.0% H_2O_2 was prepared by adding concentrated sulfuric acid and aqueous 35% hydrogen peroxide to water. When a nylon fabric having an original brightness of 73.8% was immersed in this medium at 65° C. for 2 hours, the brightness was increased to 80.5%, but a fiber strength loss of 40.4% resulted. When the bleaching time was extended to 4 hours, the final brightness was 81.7% and the fiber strength loss was 53.9%.

Example 5

A 200 g. bleaching medium containing about 6.3% H_2O_2 was prepared by adding aqueous 35% hydrogen peroxide to water along with sufficient concentrated sulfuric acid (10 to 20 drops) to reduce the pH of the resulting mixture to 1. When a nylon fabric similar to that employed in Examples 3 and 4 was immersed in this medium at 45° C. for 16 hours, the brightness increased from an original of 70.4% to a final of 76.3%.

Example 6

About 200 g. of a bleaching medium containing 1% H_2O_2 and 25% hydroxyacetic acid was prepared by adding hydroxyacetic acid and aqueous 35% hydrogen peroxide to water. The medium had a pH of 1.0 to 1.2. Nylon fabric similar to that employed in Example 5 and having a brightness of 70.4% was immersed in this medium for 16 hours at 45° C. The final brightness was 82.3%.

Example 7

A bleaching medium was prepared by adding to 196 ml. of water 0.8 ml. of aqueous 35% hydrogen peroxide and 2 ml. of a sodium silicate solution (containing the equivalent of 10% Na_2O, 25% SiO_2 and 65% H_2O). The medium had a pH of 10. Nylon fabric similar to that employed in the preceding examples and having a brightness of 72.6% was immersed in this medium for 4 hours at 85° C. This treatment gave a final brightness of 82.7% but a loss of 82.4% in fiber strength resulted.

Example 8

The general procedure of Example 1 was followed using a bleaching medium containing equal parts by weight of water and hydroxyacetic acid, the bleaching time being 16 hours and the temperature 45° C. Substantially no loss in fiber strength resulted and the final brightness was 83.1%.

Example 9

The general procedure of Example 1 was repeated using a bleaching medium containing 5% hydroxyacetic acid and the balance water. The bleaching time was 16 hours and the temperature 45° C. Substantially no loss in fiber strength resulted and the final brightness was 79.9%.

Monochloroacetic, formic and tartaric acids were found in experiments similar to those described in Examples 1 to 3 to be suitable for use in practicing the present invention.

The results of Example 4 demonstrate that when sulfuric acid is employed in place of an aliphatic carboxylic acid as a constituent of the bleaching medium, substantial improvement in brightness results but only with serious loss in fiber strength.

The results of Examples 5 and 6 show that relatively ineffective bleaching results in an aqueous bleaching medium containing no aliphatic carboxylic acid when the pH of the medium is adjusted by means of sulfuric acid to a value substantially the same as that for a medium containing 25% of hydroxyacetic acid.

Example 7 demonstrates that the usual alkaline aqueous peroxide bleaching solutions seriously degrade nylon fibers under the temperature and time conditions necessary to obtain effective bleaching with such solutions.

Hydrogen peroxide normally will be employed as the source of active oxygen in the bleaching medium, although any peroxygen compound known to be equivalent to hydrogen peroxide may be employed in place thereof. Examples of such compounds are the alkali metal peroxides, percarbonates and perphosphates, and other peroxygen compounds such as urea peroxide. The peroxygen compound may be employed in any desired amount, a concentration in the bleaching medium equal to from about 0.1 to 5% H_2O_2 being generally suitable. Concentrations in the range 0.5 to 1.5% are preferred.

I claim:

1. The method of bleaching nylon comprising subjecting a nylon material to the action of an aqueous bleaching medium having dissolved therein a peroxygen compound and at least 4%, based upon the total weight of said medium, of an aliphatic carboxylic acid having a dissociation constant in the range 1.0×10^{-3} to 1.0×10^{-6}.

2. The method of bleaching nylon comprising subjecting a nylon material to the action of an aqueous medium having dissolved therein a peroxygen compound and from 4 to 70%, based upon the total weight of said medium, of an aliphatic carboxylic acid having a dissociation constant in the range 1.0×10^{-3} to 1.0×10^{-6}.

3. The method of bleaching a nylon textile material comprising subjecting said material to the action of an aqueous medium having dissolved therein a peroxygen compound and 4 to 70%, based upon the total weight of said medium, of an aliphatic carboxylic acid having a dissociation constant in the range 1.0×10^{-3} to 1.0×10^{-6}.

4. The method according to claim 2 carried out at a temperature within the range from 35° C. to 85° C.

5. The method of claim 2 wherein the bleaching medium contains 10 to 50% of the aliphatic carboxylic acid.

6. The method of claim 2 wherein the aliphatic carboxylic acid employed has a dissociation constant in the range 1.0×10^{-4} to 1.0×10^{-6}.

7. The method of claim 2 wherein the aliphatic carboxylic acid is acetic acid.

8. The method of claim 2 wherein the aliphatic carboxylic acid is formic acid.

9. The method of claim 2 wherein the aliphatic carboxylic acid is hydroxyacetic acid.

10. The method of bleaching nylon comprising subjecting a nylon material to the action of an aqueous medium having dissolved therein a peroxygen compound and from 10 to 30%, based upon the total weight of said medium, of an aliphatic carboxylic acid having a dissociation constant in the range 1.0×10^{-3} to 1.0×10^{-6}.

11. The method of claim 10 carried out at a temperature of 35 to 85° C.

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