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PROCESS OF BLEACHING SPECIALTY HAIR FIBERS WITH HYDROGEN PEROXIDE SOLUTIONS

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This invention pertains to an improved method of bleaching proteinaceous fibers and more particularly to a method of bleaching specialty hair fibers obtained from animals other than sheep.

Specialty hair fibers represent a separate group of proteinaceous fibers. Their properties are in many respects different from the properties of proteinaceous fibers in general, and wool in particular. Some of the better known and more important specialty hair fibers are mohair, cashmere, alpaca and vicuna. The production of these fibers from the fiber bearing animals, that is, goats and camels, is very much lower than the production of true sheep wool. Low production and the peculiar characteristics of these specialty hair fibers account for the relatively high prices which these fibers command.

As will most textile fibers, the trade is particularly interested in obtaining fibers which not only possess the desired physical characteristics, such as fiber length, handle and luster, but also are of a light color. Because a large percentage of the specialty hair fibers are in dark shades of brown and even black, only a relatively small amount is available for manufacture of white garments or of garments dyed in desired light pastel colors. Even though in processing, the bulk of this dark hair is removed a certain amount is inevitably present in the bulk of lighter colored hair. When processed specialty hair fibers are bleached by procedures heretofore available, these dark hairs, which are much more difficult to bleach than the bulk of the fibers, retain much of their natural color. As a result, these hairs are very noticeable even if the bulk of the fiber is of a satisfactory light color. It is for these reasons that bleaching of specialty hair fibers is important in an attempt to make a larger volume of fiber available for white and light-colored fabrics.

Bleaching of wool is, of course, common practice and a variety of satisfactory bleaching procedures, usually involving bleaching with alkaline hydrogen peroxide solutions, are known and practiced. These procedures permit one to bleach even rather dark-colored wool to a satisfactory very light shade or even to bleach it to an almost full white. The chemical properties of the specialty hair fibers, however, are somewhat different from the properties of wool. More particularly, the specialty hair fibers are very much more sensitive to alkali. This makes it exceedingly difficult, if not impossible, to bleach the darker colored specialty hair fibers with the alkaline peroxide solutions ordinarily employed in bleaching the less alkali sensitive wool. If specialty hair fibers be bleached with the customary wool bleaching procedures, the above mentioned dark hairs remain, more or less unbleached, and thus detract from the appearance of the finished product. Yet, because of the scarcity of light specialty hair fibers, interest in bleaching them is very pronounced in the trade.

Heretofore, it was generally assumed that only those specialty hair fibers possessing a natural light color could be bleached to near white, and that available bleaching

methods were too harsh to attempt to bleach dark-colored hair fibers to truly light shades.

We have now found that under certain conditions, hereinafter to be described, even brown and very dark hair fibers can be bleached to very light shades and fully maintain the desired physical characteristics of luster, softness and handle. At the same time, the dark hair present in the bulk of the fibers is equally well bleached so that for all practical purposes the original color differences between the bulk of the fibers and the dark hairs disappear. Moreover, the improved bleaching method of this invention produces light-colored hair fibers which are particularly suitable for subsequent dyeing to light pastel shades.

Our new and improved bleaching method relies essentially on a particular way of preparing the fibers for bleaching with known peroxide bleaching baths ordinarily used for bleaching wool fibers. That is to say that the actual bleach bath used is a normal wool bleach bath. However, it is only the particular manner of preparing the fibers, in accordance with the present invention, which permits the use of such a bath with excellent results. The same bleaching bath used directly on and without the pretreatment of the specialty fibers would not result in an acceptable bleach but would rather tend to damage the fibers without substantially improving their color.

In broad outline, the method of this invention employs three separate steps. Each one of these steps is a necessary requirement and only the particular sequence of particular steps will give excellent bleaching results fully meeting the requirements of the trade.

In our method, the hair fibers are first scoured in a conventional manner, if required. They are then treated with a solution containing an iron salt and a reducing agent of an oxy acid of sulfur, wherein the sulfur has a valence of less than six. After removal from this solution, the fibers are washed and treated in combination with an acid and hydrogen peroxide. After this treatment, the fibers are again washed and subsequently bleached with a mildly alkaline peroxide bleaching bath of the type commonly used for bleaching wool.

The first treatment preferably employs a solution containing ferrous ammonium sulfate or a mixture of ferrous sulfate and ammonium sulfate, and also a reducing agent such as sodium hydrosulfite, sodium bisulfite, sodium sulfoxylate formaldehyde, zinc hydrosulfite, zinc sulfoxylate formaldehyde or other compound of an oxy acid of sulfur wherein the valence of sulfur is less than six. For reasons of cost and availability, sodium hydrosulfite will ordinarily be preferred as reducing agent. The first treatment comprises contacting the fibers for a period of time and at an elevated temperature, the time-temperature relationship being from about 10 hours at about 100° F. to 30 minutes at about 130° F., in which latter instance the solution is permitted to stand and cool for about 10 hours. The treating solution contains about 3% of either ferrous ammonium sulfate or mixtures of ferrous sulfate and ammonium sulfate, and 2% to 4% of the salt of the reducing oxy acid of sulfur.

In the second step, the fibers are treated with a mineral acid and hydrogen peroxide. Sulfuric acid is the preferred acid for reasons of cost, although hydrochloric acid or any other strong acid can be used. The function of the acid is not specific as far as the anion of the acid is concerned, so that essentially any acid will serve. The acid may be used together with or separately from the hydrogen peroxide. In other words, the fibers may be treated first with the acid and then hydrogen peroxide, or first with hydrogen peroxide and then with the acid or finally the fibers may be treated with a solution containing both the acid and the hydrogen peroxide. The second treatment is for about 1 to 2 hours at a temperature of from room temperature to 110° F. The

solution contains about 1% to 2% of the strong mineral acid and about 1.6 to 2% of 35% hydrogen peroxide, and the liquor ratio, that is the ratio of this solution to the fiber being treated is by weight about 20 parts of solution to 1 part of the fiber.

After this second step, the fibers may be neutralized if desired to reduce carry-over of acidic material into the alkaline peroxide bleach bath. Neutralization can be carried out using any alkali in dilute solution, for instance sodium carbonate, phosphates, sodium bicarbonate, etc.

The last treatment employs an ordinary wool bleaching bath containing hydrogen peroxide as the bleaching agent, and preferably a mild alkali to yield a pH of about 8. Alkalies suitable for this purpose are phosphates, such as sodium metaphosphate and sodium pyrophosphate, ammonia, etc. Combinations of alkalies may also be used, for instance combinations of sodium metasilicate and sodium bicarbonate, sodium pyrophosphate and sodium carbonate, disodium phosphate and ammonia, and others. In other words, any one of the well known mildly alkaline wool bleach baths may be used.

This sequence of steps as outlined above represents a novel and effective bleaching procedure for specialty hair fibers in general, and particularly for cashmere and alpaca fibers. However, this method may be used with equally good effects on other specialty hair fibers and on sheep wool in those instances where a particularly mild and harmless bleaching method is required fully to maintain the desirable physical characteristics of the fiber.

The following examples will illustrate the principle of this invention. In all these examples the fibers were first scoured in a conventional manner where required. All treatments described in the examples were carried out at a liquor ratio of about 20 to 1.

Example 1

A sample of dark brown cashmere was treated for about 10 hours with a solution containing 3% ferrous ammonium sulfate and 2% sodium hydrosulfite, both percentages being based on the weight of the fiber. The treatment was carried out at a temperature of about 100° F. Thereafter, the sample was washed with water and excess water removed.

In the second step the sample was immersed for about 2 hours at about 110° F. in a solution containing about 2% hydrogen peroxide and about 2% sulfuric acid. Thereafter, the sample was washed, neutralized with a solution containing about 4 g. of sodium bicarbonate per liter and washed again.

In the third step the sample was treated for about 4 hours at 110° F. with an alkaline peroxide bleach bath containing per liter of solution about 3 g. of sodium metasilicate, about 2 g. of sodium bicarbonate and about 40 ml. of hydrogen peroxide 35%. After termination of the bleaching step, the sample was washed and dried.

The originally dark brown cashmere was bleached to a very light yellowish color and the dark hairs present originally were bleached to essentially the same color. Luster and softness of the fibers were fully retained.

Example 2

A sample of dark brown alpaca was treated in a first step in the manner described in Example 1 but the treating solution contained, based on the weight of the fibers, about 2% of ferrous sulfate, about 1% of ammonium sulfate and about 3% of sodium hydrosulfite. Also added was about 1% of a commercial wetting agent.

After this first step, the sample was processed exactly as described in Example 1.

The originally dark brown alpaca was bleached to a very light yellowish color and the dark hairs present originally were bleached to essentially the same color. Luster and softness of the fibers were fully retained.

Example 3

A sample of black caracul wool was treated in steps 1 and 2 as described in Example 1. It was then bleached as described in Example 1 but with the use of a bleach bath containing about 2 g. per liter of tetrasodium pyrophosphate, about 2 g. per liter of sodium carbonate and about 40 ml. per liter of hydrogen peroxide 35%.

The originally black caracul was bleached to a very light yellowish color and the dark hairs present originally were bleached to essentially the same color. Luster and softness of the fibers were fully retained.

Example 4

In this example, dark brown cashmere was treated as described in Example 1 but the bleach bath employed as the third step contained per liter about 2 g. of disodium phosphate and about 30 ml. of hydrogen peroxide 35%, and was adjusted to about pH 8 by the addition of a small amount of ammonium hydroxide.

The originally dark brown cashmere was bleached to a very light yellowish color and the dark hairs present originally were bleached to essentially the same color. Luster and softness of the fibers were fully retained.

Example 5

A sample of dark brown cashmere was treated exactly as described in Example 1 but the treatment of the second step was carried out by first immersing the sample in a solution containing approximately 2% of hydrogen peroxide 35% and after 1 hour treatment at room temperature, approximately 2% of sulfuric acid was added to the bath and treatment continued for another hour.

After bleaching with an alkaline peroxide bleach bath as described in Example 1, the originally dark brown cashmere was bleached to a very light yellowish color and the dark hairs present originally were bleached to essentially the same color. Luster and softness of the fibers were fully retained.

Example 6

A sample of dark brown alpaca was treated as described in Example 1 but, in the second step, the fibers were first treated by immersion for 1 hour at room temperature in a solution containing about 2% of hydrochloric acid. Thereafter about 40 ml. per liter of hydrogen peroxide 35% were added to the solution and the treatment continued for another hour.

The originally dark brown alpaca was bleached to a very light yellowish color and the dark hairs present originally were bleached to essentially the same color. Luster and softness of the fibers were fully retained.

Example 7

100 pounds of dark-colored cashmere was treated with a solution containing 1% of a sulfated alcohol type wetting agent, 3% of ferrous ammonium sulfate and 4% of sodium hydrosulfite. The treatment was carried out for 30 minutes at a temperature of about 130° F. and the goods were then permitted to rest in the solution for about 10 hours. Thereafter, the goods were washed with water. The above percentages are based on the weight of goods treated.

In the second step the goods were treated for 1 hour at 110° F. with a solution containing about 1.6% of hydrogen peroxide 35%. Thereafter, 1% of concentrated sulfuric acid was added to that solution and treatment continued for an additional 30 minutes at the same temperature. Thereafter, the goods were washed with water.

In the third step the goods were treated with an alkaline peroxide bleach bath containing 0.47% of sodium bicarbonate, 0.35% of tetrasodium pyrophosphate and 3.5%

of hydrogen peroxide 35%. This treatment was carried out for two hours at 125° F. Finally, the goods were washed with water.

The originally dark brown cashmere was bleached to a very light yellowish color and the dark hairs present orignally were bleached to essentially the same color. Luster and softness of the fibers were fully retained.

As remarked above, the process of this invention is critical only as far as the particular sequence of the three particular steps is concerned. These steps are:

First, treatment with iron salt and reducing agent.

Second, treatment with acid and hydrogen peroxide in combination.

Third, treatment with alkaline peroxide bleach bath.

The exact conditions of treatment in each step are not critical in the sense of this invention and are dictated only by the known physical and chemical characteristics of the fibers. This is particularly true as far as temperature of treatment, liquor ratio, time and auxiliary procedures, such as washing or neutralizing are concerned.

What is claimed is:

1. Method of bleaching a specialty hair fiber which is a member of the group consisting of mohair, cashmere, alpaca, vicuna, and camel hair fibers, which comprises contacting said fibers for a period of time and at an elevated temperature, said time-temperature relationship being from about 10 hours at about 100° F. to 30 minutes at about 130° F., in which latter instance the solution is permitted to stand and cool for about 10 hours, with an aqueous solution containing on the weight of said fibers about 3% of ferrous ammonium sulfate and about 2–4% of a water-soluble salt of a reducing oxy acid of sulfur wherein the sulfur has a valence of at least 2 but less than 6, separating said fibers from said solution and washing them with water, contacting said separated and washed fibers for about 1–2 hours and at about room temperature to 110° F. with about 20 parts by weight of an aqueous solution to one part by weight of the fibers, said solution containing about 1–2% of a strong mineral acid and about 1.6–2% of 35% hydrogen peroxide, said acid and said hydrogen peroxide being in dilute aqueous solution, separating said fibers from said aqueous solution, and thereafter treating said separated fibers with a bleaching amount of an aqueous solution of hydrogen peroxide at a pH of about 8 until said bleaching has been completed.

2. Method of bleaching a specialty hair fiber which is a member of the group consisting of mohair, cashmere, alpaca, vicuna, and camel hair fibers, which comprises contacting said fibers for a period of time and at an elevated temperature, said time-temperature relationship being from about 10 hours at about 100° F. to 30 minutes at about 130° F., in which latter instance the solution is permitted to stand and cool for about 10 hours, with an aqueous solution containing on the weight of said fibers about 3% of ferrous ammonium sulfate and about 2–4% of a water-soluble salt of a reducing oxy acid of sulfur selected from the group of reducing acids consisting of sulfurous acid, sulfoxylic acid and hydrosulfurous acid, separating said fibers from said solution and washing them with water, contacting said separated and washed fibers for about 1–2 hours and at about room temperature to 110° F. with about 20 parts by weight of an aqueous solution to one part by weight of the fibers, said solution containing about 1–2% of strong mineral acid from the group consisting of sulfuric acid and hydrochloric acid and about 1.6–2% of 35% hydrogren peroxide, said acid and said hydrogen peroxide being in dilute aqueous solution, separating said fibers from said aqueous solution and thereafter treating said separated fibers with a bleaching amount of an aqueous solution of hydrogen peroxide at a pH of about 8 until said bleaching has been completed.

3. Method of bleaching a specialty hair fiber which is a member of the group consisting of mohair, cashmere, alpaca, vicuna, and camel hair fibers which comprises contacting said fibers with an aqueous solution containing on the weight of said fibers about 3% of ferrous ammonium sulfate, and about 2–4% of a water-soluble salt of a reducing oxy acid of sulfur wherein the sulfur has a valence of at least 2 but less than 6, said solution being at about 130° F. for about thirty minutes and thereafter being permitted to reach its own temperature level for about 10 hours, separating said fibers from said solution and washing them with water, contacting said separated and washed fibers for about 1–2 hours and at about room temperature to 110° F. with about 20 parts by weight of an aqueous solution to one part by weight of the fibers, said solution containing about 1–2% of a strong mineral acid and about 1.6–2% of 35% hydrogen peroxide, said acid and said hydrogen peroxide being in dilute aqueous solution, separating said fibers from said aqueous solution, and thereafter treating said separated fibers with a bleaching amount of an aqueous solution of hydrogen peroxide at a pH of about 8 and at a temperature of about 110° F. to 125° F., until said bleaching has been completed.

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