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PROCESS FOR BLEACHING UNBLEACHED CELLULOSE

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ABSTRACT OF THE DISCLOSURE

Unbleached cellulose is bleached to a good degree of whiteness with low degradation and with removal of any size or cotton husk present, by a continuous process involving successive contact with (1) amylase, (2) a compound such as trichloroisocyanuric acid containing a nitrogen-chlorine linkage, and (3) an oxidative bleaching agent.

This invention relates to the bleaching of unbleached cellulose.

Any satisfactory process for bleaching unbleached cellulose textile materials, especially those that contain synthetic fibers, must not only bleach the cellulose, but must also remove any cotton husks any size which may be present, increase the hydrophilicity of the fiber, and not lead to any substantial degradation of the cellulose molecules. Certain enzymatic materials, such as amylases, are known effective desizing agents for textile materials, but have no bleaching effect, and do not remove cotton husks, nor improve the hydrophilicity of the fibers. Conversely, conventional bleaching agents such as hydrogen peroxide and sodium chlorite have a good bleaching effect, but do not remove size and, in many cases, do not improve the hydrophilicity of the fibers. The hydrophilicity can be improved by boiling with highly concentrated sodium hydroxide solution, but this treatment tends to degrade the cellulose, and in any case involves the use of an additional process step. At the present time, it is generally necessary in industrial practice to subject the cellulose material to be bleached to a succession of different treatments which are applied discontinuously and which require large apparatus and a considerable amount of time. There is therefore a need for a process which can be operated continuously at high speed which not only bleaches cellulosic textile fabrics, but also removes any cotton husks and size which may be present, increases the hydrophilicity of the fibers, and does not degrade the cellulose to any substantial degree.

The present invention provides such a continuous process for bleaching unbleached cellulose, which process comprises contacting the said unbleached cellulose with, in the following order, (1) amylase, (2) a compound containing one or more nitrogen-chlorine linkages, and (3) a conventional oxidative bleaching agent. This new process can be operated continuously at high speed, e.g. at a feed rate of cellulose textile material of 50 to 100 meters per minute. Surprisingly, the combination of treatments thus carried out gives results which are better than the sum of the results obtained by each treatment carried out separately. In particular, the treatment (2) with the compound containing one or more nitrogen-chlorine linkages activates the subsequent action of the bleach, thereby reducing to a substantial degree the time required for the latter to have its desired effect. This is very important industrially, as in prior continuous processes it was necessary to store, e.g. in a J-box, the fabric impregnated with the bleach at 100° C. for a considerable period, e.g. an hour. Thus, if the fabric moves at a 100 meters per minute, 6,000 meters of fabric, which may weigh more than a ton, can be held up in the J-box. While it is possible to reduce the time required for the bleach to act by adding activators to the bleach, these have the effect of seriously reducing its effective life and of making the bleaching effect difficult, or impossible, to control adequately.

In the process of the invention, the compound containing one or more nitrogen-chlorine linkages is, for example, an N-chlorinated organic compound such as N-chlorotoluene-sulphonamide or more advantageously, a chlorinated cyanuric acid. This compound is applied from an aqueous bath having a pH of 4 to 8 and a content of available chlorine from 2 to 20 grams per liter.

While the conventional bleach may be any commercially used bleaching agent, e.g. an alkali metal chlorite or hypochlorite, best results have been obtained by the use of hydrogen peroxide.

The process of the invention can, if desired, be followed by a treatment of the cellulose in an alkaline bath, and it is an advantage of the new process that this treatment may be very short so that the cellulose material is not degraded to any substantial extent.

In more detail, the new process can be carried out as follows. The cellulose textile material, e.g. in the form of threads, tows, or woven fabric, is passed into a conventional castor-tank containing an aqueous solution of commercial amylase having a concentration of 2 to 10 grams per liter and at a temperature from 20 to 80° C. This bath can also contain 1 to 2 grams per liter of a compatible wetting agent. The material remains in this bath for from 5 to 30 seconds, depending upon the concentration of the amylase. It is then squeezed to 50 to 120% wet take-up, washed with boiling water, and squeezed to 50 to 70% wet take-up. The cellulose material is passed to a second conventional castor-tank containing the chlorinated derivative of cyanuric acid (or other compound containing a nitrogen-chlorine linkage) in a concentration giving an available chlorine content of 2 to 20 grams per liter, at a temperature of 10 to 60° C. and a pH of 4 to 8. The cellulose material can remain in this tank for from 5 to 30 seconds, and be fed at a speed of up to 100 meters per minute, so that the tank can ordinarily contain from, say, 10 to 30 meters of fabric. After leaving the second bath, the fabric is washed with hot water and squeezed.

The consumption of amylase in the first bath is generally between 0.3 and 0.5%, and that of the chlorine-containing compound in the second bath between 0.1 and 0.8%, the percentages being by weight of the material being treated.

After the aforesaid washing with hot water and squeezing, the cellulose fabric is fed to a tank containing hydrogen peroxide stabilized with sodium silicate, or any other suitable bleach. After leaving this bath, it is squeezed to 60 to 100% wet take-up and then kept for ½ to 10 minutes in a J-box or a steamer at 100° C. or for ½ to 1 minute in a steamer under pressure at 130 to 140° C.

The material is then washed at about 100° C. in sodium hydroxide solution having a concentration of 5 to 10 grams per liter, and finally washed carefully and dried.

The concentration of hydrogen peroxide is from 1 to 3% by weight, expressed as 130 volume (35%) hydrogen peroxide. This is approximately the same concentration as is prior processes. However, if it desired to reduce the treatment time with hydrogen peroxide to ½ to 2 minutes at a temperature of 100° C., the concentration of hydrogen peroxide in the treatment bath must be increased to 4 to 5%.

An alkali metal chlorite, e.g. sodium chlorite, may be substituted for the hydrogen peroxide. If this is

done, it is necessary before the fabric is immersed in the chlorite bath to wash it very thoroughly to remove any excess of chloro-cyanuric compound which would react with the chlorite and cause decomposition of the latter. A suitable chlorite concentration is 10 to 30 grams per liter, and the bath can contain a conventional adjuvant such as sodium phosphate. The fabric is squeezed to 80 to 100% wet take-up and then kept at a 100° C. for 5 to 15 minutes before being washed and dried. No chlorine dioxide is formed during the bleaching and the product obtained is completely free from husks and stains and has a good degree of whiteness and excellent hydrophilicity.

The degree of hydrophilicity imparted by a bleaching process may be estimated by measuring the time required for a disc of fabric 20 mm. in diameter to become totally immersed in water at 20° C. after being laid on the surface. For example raw cotton satin bleached in a bath containing 3% of 130-volume hydrogen peroxide (based on the weight of cotton) for 10 minutes at 100° C., after washing and drying, takes longer than 5 minutes to become totally immersed. When treated in accordance with the present invention, and bleached in a bath containing only 2% of 130-volume hydrogen peroxide for 10 minutes at 100° C. followed by washing and drying, the same fabric has an immersion time of only 2 seconds. Moreover, if the treatment with the chloro-cyanuric compound is omitted, the preliminary treatment with amylase gives only a relatively insignificant improvement in hydrophilicity.

The following examples illustrate the invention.

EXAMPLE 1

A starch-sized, cotton poplin fabric 140 centimeters in width and weighing 200 grams per meter was subjected to the process of the invention without previous desizing. The fabric was fed to a castor-tank having a capacity of 20 meters of the fabric at a rate of 80 meters per minute. The bath contained 5 grams of amylase, 10 grams of sodium chloride, and 1 gram of a wetting agent based on the octylester of sulphosuccinic acid, per liter. The temperature of the bath was 70° C. and the immersion time 15 seconds. The fabric was squeezed to a 70% wet take-up after leaving the tank and then fed to a washing tank containing water at 100° C. and having a capacity of 15 to 20 meters of the fabric. Amylase starch size were removed by this washing. The fabric was then squeezed to a 55% wet take-up and fed to a second castor-tank containing a solution at 30° C. of 8.25 grams per liter of trichloroisocyanuric acid, 2.29 grams per liter of cyanuric acid, and 4.46 grams per liter of sodium bicarbonate. The fabric remained in this tank for 15 seconds and was then squeezed to 70% wet take-up and passed to a washing tank containing water at 100° C., where it was washed for 10 seconds and the excess isocyanuric acid derivative was removed. The fabric was then squeezed to 55% wet take-up and passed to a third tank, it was soaked in a solution at 30° C. containing 15 grams per liter of sodium hydroxide, 25 grams per liter of 36° Bé. sodium silicate solution, 7 grams per liter of a wetting agent based on a condensate of a fatty acid with a depolymerized protein, and 30 milliliters of 35% aqueous hydrogen peroxide. The concentration of this bath was kept constant by adding to it as necessary a solution containing the same ingredients in triple concentration. The fabric after leaving this bath was squeezed to a 75% wet take-up and then passed vertically through a steamer at 100° C. It was then fed to a maturing chamber having a capacity of 1000 meters and kept therein for 10 minutes at 100° C. The fabric was then passed to a series of five washing tanks, the first of which contained sodium hydroxide solution in a concentration of 10 grams per liter at 100° C. and the other four hot water.

Very white, completely desized fabric of excellent hydrophilicity was obtained. The polymerization degree of the cellulose was reduced only from 2700 for the unbleached cotton to 2100 for the bleached fabric.

EXAMPLE 2

A starch-sized cream colored mixed fabric having a cotton warp and a thread-bleached linen woof weighing 250 grams per square meter was subjected to the treatment described in Example 1 at a rate of 50 meters per minute. The first tank contained 5 grams per liter of amylase, 10 grams per liter of sodium chloride, and 2 grams per liter of non-ionic wetting agent. Its temperature was 60° C. The bath containing the isocyanuric acid derivative had a temperature of 40° C. and a concentration 50% higher than that described in Example 1. The bleaching with hydrogen peroxide was carried out under the conditions described in Example 1.

The fabric obtained had a degree of whiteness corresponding to ¾ white, was perfectly desized, and had a very good degree of hydrophilicity. The polymerization degree was reduced from 2100 to 1800. The non-cellulosic parts of the linen and the cotton husks were completely removed.

In this example, or in any of the other examples, the amylase employed can be any commercially available amylase or amylase preparations known as a textile desizing agent. Thus, use can be made of the commercially available Rapidase, a known textile desizing agent.

EXAMPLE 3

The fabric treated was a mixed polyester/cotton poplin intended for a resin treatment combined with waterproofing. The poplin weighed 120 grams per square meter and had a width of 90 centimeters. The treatment with amylase was carried out at 70° C. for 20 seconds in a bath containing 7 grams per liter of Rapidase, 10 grams per liter of sodium chloride, and 1 gram per liter of a wetting agent based on an alkyl-aryl sulphonate. After washing, the poplin was contacted for 20 seconds with a bath at 20° C. containing 10 grams per liter of sodium dichloroisocyanurate containing 60% of available chlorine. Washing and bleaching with hydrogen peroxide were carried out as in Example 1, the maturing time being only 5 minutes at 100° C.

The treated fabric was completely desized and had a high degree of whiteness. The degree of polymerization of the cellulose was reduced from 2580 to 2050 and the hydrophilicity was such that the immersion time of a fabric disc was 1 second.

EXAMPLE 4

A cotton twill fabric intended for vat dyeing on a jigger was treated with amylase and a chlorocyanuric acid derivative as described in Example 1 and, after washing, was soaked in a solution containing 20 grams per liter of sodium hydroxide, 20 grams per liter of 36° Bé. sodium silicate solution, 5 grams per liter of a polymeric sodium metaphosphate, and 50 milliliters of 35% aqueous hydrogen peroxide solution. After squeezing to 100% wet take-up, the fabric was rolled up and rotated slowly for 10 hours at 20° C. The fabric was then unfolded and washed. The husk and starch had been completely removed. The fabric had a sufficient degree of whiteness for dyeing and very good hydrophilicity. The polymerization degree of the cellulose was reduced from 2500 to 2100.

EXAMPLE 5

A starch-sized mixed cotton/polyamide fabric weighing 80 grams per square meter and 80 centimeters in width was soaked in a castor-tank having a capacity of 10 meters of fabric and containing 5 grams per liter of amylase, 8 grams per liter of sodium chloride, and 2 grams per liter of non-ionic wetting agent at a rate of 100 meters per minute. After squeezing to 60% wet take-up, the fabric was fed to a washing tank and again

squeezed to 60% wet take-up. The fabric was then fed to a second castor-tank at 40° C. containing 12.4 grams per liter of trichlorocyanuric acid, 3.3 grams per liter of cyanuric acid, and 6.7 grams per liter of sodium bicarbonate. The fabric was squeezed to 60% wet take-up and then passed unfolded into a washer having 3 tanks wherein the excess of the chlorocyanuric acid derivative was washed out with very hot water. The fabric was then bleached by feeding into a tank containing 15 grams per liter of 100% sodium chlorite, and 3.6 grams per liter of monosodium phosphate. The fabric was squeezed to 65% wet take-up and fed to a steamer at 100° C. The fabric was folded in a maturing vessel where it was kept for 5 minutes at 100° C. When bleaching was completed, the fabric was fed unfolded into a washer having four tanks, the first containing 10 grams per liter of sodium hydroxide and the other three very hot water. Fabric having good hydrophilicity and very high degree of whiteness was obtained. All the size was removed and the mechanical strength of the fabric was reduced only by 5% compared with the unbleached fabric.

What is claimed is:

1. Continuous process for bleaching unbleached cellulose textile material which comprises contacting the said material with, in the following order:

(1) an aqueous solution of amylase having a concentration of 2 to 10 grams per liter at a temperature from about 20 to 80° C.

(2) an aqueous solution of chlorocyanuric compound containing 2 to 20 grams per liter of available chlorine having a pH of 4 to 8 at a temperature from about 10 to 60° C., and

(3) an oxidative bleaching agent which is hydrogen peroxide or sodium chlorite.

2. A process according to claim 1 wherein the chlorocyanuric compound is sodium dichloroisocyanurate or trichloroisocyanuric acid.

3. A process according to claim 1 wherein the said unbleached material is contacted for 5 to 30 seconds with the chlorocyanuric compound.

4. A process according to claim 1 wherein the said unbleached material is contacted for 5 to 30 seconds with the aqueous solution of amylase.

5. A process according to claim 1 wherein the oxidative bleaching agent is hydrogen peroxide in the form of an aqueous solution containing 1 to 3% by weight of 130 volume (35%) hydrogen peroxide, and the contact time is ½ to 10 minutes.

6. A process according to claim 1 wherein the oxidative bleaching agent is soduim chlorite in the form of an aqueous solution having a concentration of 10 to 30 grams per liter, and the contact time is 5 to 15 minutes.

7. A process according to claim 1 wherein the cellulose textile material is thoroughly washed before being contacted with the chlorocyanuric compound and before being contacted with the oxidative bleaching agent.

8. A process according to claim 1 wherein the cellulose textile material is subjected to an alkaline wash after treatment with the oxidative bleaching agent.

9. A process according to claim 1 wherein the cellulose textile material is in the form of a continuous fabric which is treated continuously at a rate of at least 50 meters per minute.

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