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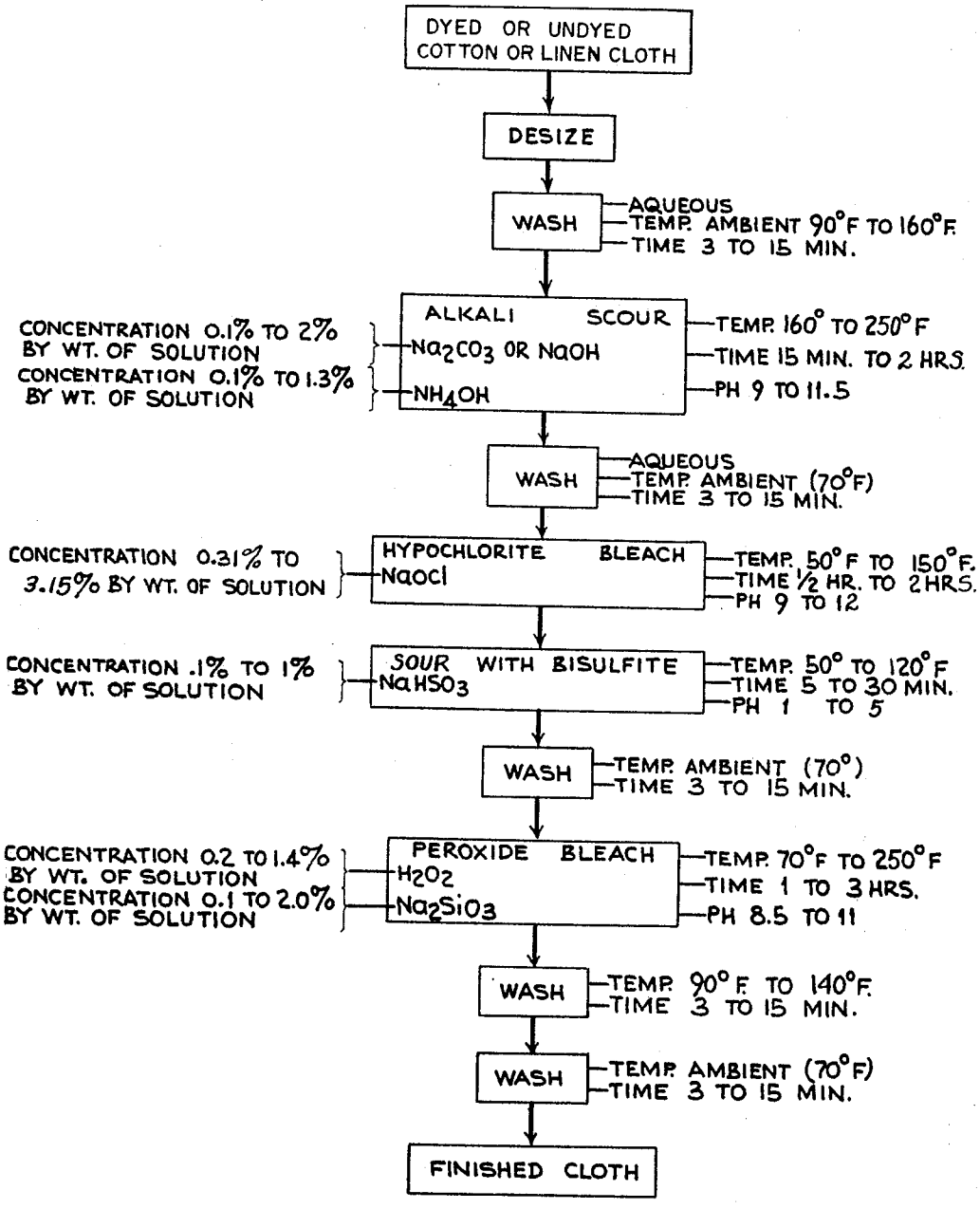

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PROCESS OF SCOURING AND BLEACHING DYED AND UNDYED CELLULOSE TEXTILES

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This application is a continuation-in-part of our copending application Serial No. 65,862 filed October 28, 1960 and now abandoned.

The present invention relates to the treatment of textiles. More particularly, the present invention relates to the bleaching of bast fibers and cloth made from these fibers, such as linen. In addition, the present invention relates to the bleaching of cotton and linen cloth containing colored ingrain patterns and/or stripes.

In the bleaching of linen cloth and fibers, it is a most difficult problem to acquire adequate brightness of the material treated without seriously interfering with other desirable qualities of the cloth or fiber treated. Thus, frequently, high brightness achieved during bleaching results in fiber or cloth having tensile strengths considerably below accepted standards. In addition, it is often found that the acquisition of suitable tensile strength in a bleached linen fiber or cloth results in the concurrent production of a cloth or fiber having inadequate brightness qualities. Still further, in the bleaching of cellulosic goods such as cotton and linen goods that contain portions which have been dyed with dyes not completely fast to conventional peroxide bleaching solutions, as for instance, naphthol, indigo, anthraquinoid and other similar dyes, bleaching processes which achieve adequate degrees of brightness for commercial standards result in the bleeding of this predyed stripe or pattern. Processes involving bleaching linen cloth also require bleaching sequences which are quite time consuming, often necessitating operations lasting forty hours or more.

In accordance with the present discovery, many of these difficulties facing the prior art have been eliminated. Thus, linen cloth and fibers treated in accordance with the teachings of this invention are found to attain exceptionally high degrees of brightness while maintaining tensile strengths well within accepted standards. Similarly, cotton and linen materials containing predyed striping or patterns with dyes not fast to usual peroxide bleaching solutions, are now capable of being bleached to a high degree of brightness while maintaining adequate tensile strength and with considerably less bleeding of the dye occurring. Still further, when applied to the treatment of linen cloth, absorbency of the finished product is considerably improved, thus rendering the process extremely desirable in applications involving linen toweling. In addition, substantial reductions in the time required to bleach linen cloth are readily achieved.

According to the present invention, linen cloth which has been treated by conventional desizing operations is passed through two alkaline baths which scour and bleach the goods. After the second alkaline treatment, the goods are soured with a sodium bisulfite solution and then treated in a hydrogen peroxide bleaching bath. The sequential treatment of the linen cloth in two alkaline baths and the final hydrogen peroxide bleaching solution gives rise to linen material having exceptionally high degrees of brightness and good tensile strength. It is found that linen goods treated in this manner also attain absorbent qualities not usually found in linen materials having tensile strengths on the order of those found in material treated in this manner.

In the sequential alkaline bath treatments, the goods

are subjected in the first bath to an alkaline solution containing small quantities of ammonium hydroxide. The alkaline solution is an aqueous solution of sodium carbonate or sodium hydroxide or a combination of the two. In this bath the goods are boiled for periods of time ranging from fifteen minutes upwards. After completion of the initial boiling step, the goods are passed to a hypochlorite bleaching bath maintained at a pH of 9 to 12. Treatment in this latter bath is usually conducted at room temperature and for a period of time ranging from one-half hour upwards. After completion of the hypochlorite bleaching step, the goods are passed into a bath containing sodium bisulfite and maintained in an acid pH range of between 1 and 5. From this sodium bisulfite solution, the goods are passed into a hydrogen peroxide bleaching tank where they are bleached to final whiteness.

For a more complete understanding of the present invention, reference is made to the flow sheet and the following details which describe in a more precise manner the various conditions to be employed in the washing and treating steps of the instant process.

The desizing of the cotton and/or linen cloth treated may be conducted in any conventional enzyme desizing solution such as, for example, an amylase enzymatic bath. Baths of this kind and the treatment steps utilized to accomplish desizing are well known in the art and exemplary baths and treatments are described in "Chemistry and Chemical Technology of Cotton," edited by Kyle Ward, Jr., published by Interscience Publishers, Inc., N.Y. 1955, pages 134 through 144. Upon completion of the desizing operation, the cloth is subjected to an aqueous wash in warm water at a temperature in the range of from 90° F. to 160° F. for periods from three to fifteen minutes. Upon completion of the washing operation, the cloth is then introduced into the first stage of the bleaching process comprising the instant invention.

The first alkaline treatment the cloth is subjected to involves the contact of the cloth with an aqueous alkaline solution of sodium carbonate or sodium hydroxide containing ammonium hydroxide. The sodium carbonate or sodium hydroxide concentration in this aqueous alkaline solution is usually maintained below 2 percent by weight of solution. Normally, the sodium carbonate or sodium hydroxide concentration of the solution is about 0.5 percent by weight, but may range broadly from between 0.1 percent up to about 2 percent. Ammonium hydroxide concentrations in the alkaline bath are maintained in a range of from 0.1 percent up to 1.3 percent. Generally, the ammonium hydroxide concentration is maintained somewhere between 0.8 percent and 1.1 percent by weight. During the treatment conducted in the aqueous alkaline solution containing ammonium hydroxide, the aqueous solution is maintained at a pH in the range of between 9 and 11.5. Temperatures of the solution are maintained on the warm side and range generally between 160° F. to 250° F. The lengths of time the cloth is maintained in contact with the solution may be varied somewhat, but generally is between fifteen minutes to two hours. During the contact of the cloth with the aqueous alkaline solution, the cloth is essentially saturated with solution. Material after treatment in the aqueous alkaline bath is removed and washed in a cold water wash for a period of from three to fifteen minutes.

After the washing step, the goods are then introduced into a hypochlorite bleaching tank. The hypochlorite bleaching tank contains an aqueous solution of hypochlorite, usually sodium hypochlorite, but alkaline earth metal hypochlorites may also be employed if desired. Preferably, sodium hypochlorite is utilized in this bleaching bath. The hypochlorite concentration in the aqueous hypochlorite bleaching bath is maintained in a range of from 3 to 30 grams per liter hypochlorite measured as

available chlorine. Stated in another way, the hypochlorite concentration in the aqueous hypochlorite bleaching solution is maintained in a range of from 0.31 to 3.15 percent by weight. During contact and treatment in the hypochlorite bleaching bath, the goods are maintained for periods of time ranging between thirty minutes and two hours, and at temperatures ranging broadly between 50° F. to 150° F. Preferably, the temperature of the hypochlorite bleaching tank is maintained somewhere between 65° F. and 95° F. The goods in the hypochlorite bleaching tank are essentially saturated with the hypochlorite solution.

Goods, after treatment in the hypochlorite tank, are passed into a souring tank containing sodium bisulfite in concentrations ranging from 0.1 percent by weight of solution up to 1 percent by weight of solution. The souring conducted in the sodium bisulfite aqueous solution is usually conducted at room temperatures, but the temperature may vary between 50° F. to 120° F. Treatment in this batch is usually quite short and ranges anywhere from five minutes to one-half hour. Upon completion of the souring in the sodium bisulfite souring bath, the goods are washed in cold water usually at ambient temperatures for periods of from three to fifteen minutes.

Upon completion of this latter wash, the goods are then introduced into an aqueous hydrogen peroxide bleaching solution having a pH between 8.5 and 11 containing between 0.2 and 1.4 percent by weight of hydrogen peroxide (anhydrous) basis the weight of the aqueous solution. Also present in the hydrogen peroxide bleaching solution are small quantities of sodium silicate. Sodium silicates which may be employed have the general formula $Na_2O(SiO_2)_x$, where x represents a number from 2 to 4. The concentration of the sodium silicate contained in the hydrogen peroxide bleaching bath is in a range generally between 0.1 and 2.0 percent by weight of solution. Temperatures in the hydrogen peroxide bleaching bath are maintained generally between 70° F. and 250° F., but are usually maintained within the range of between 60° F. to 185° F. The goods are treated in the hydrogen peroxide bleaching bath for periods of time ranging between one hour and three hours.

Upon completion of the bleaching cycle in the hydrogen peroxide bleaching bath, the goods are subjected to a double wash, the initial wash consisting of a warm water wash at temperatures from 90° F. to 140° F. for periods of between three and fifteen minutes. Upon completion of the warm water wash, the goods are then passed into a cold water wash tank and subjected to cold water washing at ambient temperatures of 70° F. for periods of from three to fifteen minutes. Upon completion of the final cold water wash, the goods are then ready for drying, pressing and final processing.

For more complete understanding of the present invention, reference is made to the following examples which are indicative of the modes of operation which may be employed in conducting the process of the instant invention.

EXAMPLE I

A desized piece of linen sheeting having a colored ingrain pattern was placed in a scouring bath containing 3.74 liters of solution, having 1.5 percent NH_4OH by weight, 1 percent Na_2CO_3 by weight, and 0.1 percent Sandopan DTC (a sulfonated alcohol wetting agent manufactured under the trade name "Sandopan DTC" by the Sandoz Chemical Corporation, Incorporated). The scouring bath was contained in a laboratory apparatus designed to simulate a commercial jig. The linen material was rotated continuously in the jig and was padded on the return side of the cycle and run at 200° F. for a period of thirty minutes. Upon completion of the thirty minute contact of the solution with the cloth, the solution was discharged from the machine and the material washed with cold water at ambient temperature (70° F.).

A second aqueous solution of a 3.75 liter volume was prepared and contained 1.5 percent NaOCl by weight. The solution was introduced into the jig and the material rotated as before for a period of one hour at 95° F. Upon completion of this treatment, the solution was discharged and the material rinsed with cold water at 70° F.

A third solution of 3.74 liters volume was made up containing 0.1 percent sodium bisulfite by weight of solution. This solution was introduced into the apparatus and contacted with the cloth contained therein for a period of ten minutes at 70° F. Upon completion of this step, the solution was discharged from the apparatus and the material given a rinse in cold water at 70° F. A final aqueous solution of 3.74 liters by volume and containing 2 percent H_2O_2 (35 percent) and 0.5 percent Na_2SiO_3 was placed in the apparatus. The material was rotated with the solution at 180° F. for a period of two hours. This solution was then discharged and the material washed with hot water at 140° F. for a period of five minutes, and washed a second time with a cold water wash at 70° F. for a further period of five minutes. The material treated was designated as Sample No. 1.

EXAMPLE II

Utilizing the procedure of Example I and the solutions, times and temperatures remaining the same, a piece of cotton sheeting having a colored ingrain pattern was placed in the laboratory apparatus and subjected to the same treatment as the linen cloth of Example I. The cloth so treated was designated as Sample No. 1A.

EXAMPLE III

Utilizing the solutions identical with those prepared in Example I, a sample of linen sheeting having a colored ingrain pattern and a sample of cotton sheeting having a colored ingrain pattern were subjected individually to the identical bleaching sequence of Example I, but all operations were conducted in a Gaston County Package dyeing machine. The linen sample obtained in this example was designated No. 2 and the cotton sample was designated No. 2A.

The samples obtained from Examples I, II and III were subjected upon completion of the bleaching steps to tests for brightness, absorbency and tensile strength. Brightness was determined with a Hunter multipurpose reflectometer. Absorbency was measured by dropping a drop of water from a pipe held 2 inches from the surface of the unstretched cloth. The time required for the disappearance of the specular reflectance from a drop, as visually observed, is a measure of the absorbency.

Table I

Sample No.	Reflectance Blue	Tensile Strength, lbs./in.	Absorbency, sec.
68255-178-			
1	70.2	88.5	1.0
1A	84.3	62.7	1.0
2	69.7	82.5	1.0
2A	85.4	58.2	1.0

No bleeding of the colored ingrain pattern was observed in the finished product.

EXAMPLE IV

A desized piece of linen sheeting having a colored ingrain pattern is placed in a scouring bath containing 3.74 liters of solution, having 1.5 percent NH_4OH by weight, 1 percent NaOH by weight and 0.1 percent by

weight Sandopan DTC (a sulfonated alcohol wetting agent manufactured under the trade name "Sandopan DTC" by the Sandoz Chemical Corporation, Incorporated). The scouring bath is contained in a laboratory apparatus designed to simulate a commercial jig. The linen material is rotated continuously in the jig and is padded on the return side of the cycle and is run at 200° F. for a period of 30 minutes. Upon completion of the thirty minute contact of the linen with the solution, the solution is discharged from the machine and the linen material is then washed with cold water at ambient temperature (70° F.).

A second aqueous solution of 3.75 liters volume is prepared containing 1.5 percent NaOCl by weight. The solution is introduced into the jig and the material is rotated as before for a period of one hour at 95° F. Upon completion of this treatment, the solution is discharged and the material is rinsed with cold water at ambient temperature (70° F.).

A third solution of 3.74 liters volume is made up containing 0.1 percent sodium bisulfite by weight. This solution is introduced into the apparatus and is contacted with the cloth contained therein for a period of ten minutes at 70° F. Upon completion of this step, the solution is discharged from the apparatus and the material is given a rinse in cold water at 70° F.

A final aqueous solution of 3.74 liters by volume and containing 2 percent H_2O_2 (35 percent by weight) and 0.5 percent Na_2SiO_3 is placed in the apparatus. The material is rotated with the solution at 180° F. for a period of two hours. This solution is then discharged and the material is washed with hot water at 140° F. for a period of five minutes and is washed a second time with a cold water wash at 70° F. for a further period of five minutes.

After drying the treated linen is tested for brightness and absorbency. The cloth is found to have a high reflectance in excess of 84 as measured on a Hunter reflectometer and an absorbency of 1 second. No bleeding of the colored ingrain pattern is observed.

As can be readily seen from the above table, linen cloth treated in the manner outlined hereinabove attains and sustains a high degree of brightness with very good tensile strength. In addition, cotton cloth treated in the above manner also is adequately bleached while attaining adequate tensile strength and good absorbency.

While the invention has been described with reference to cotton and linen goods composed essentially of cotton or linen fibers, it is of course to be understood that goods treated are intended to include fabrics and cloths which are composed of mixtures of cotton fibers and/or linen fibers with other fibers such as wool, rayon, nylon, and other synthetic and natural fibrous materials easily blended with cotton and/or linen. Cloth unions, as contemplated generally, contain at least 15% by weight of cotton and/or linen therein, and include cloth which has been woven as well as knitted cloth.

While the invention has been described with reference to certain specific examples, it is not intended to be so limited except insofar as appears in the accompanying claims.

We claim:

1. A method for bleaching textiles of the group consisting of cotton and linen comprising scouring the textiles in an aqueous solution of from 0.1 to about 2.0 percent by weight of the solution of a member selected from the group consisting of sodium carbonate and sodium hydroxide, and ammonium hydroxide in a concentration of from 0.1 to 1.3 percent by weight of the solution, having a pH of between 9 and 11.5, at a temperature of between 160° F. to 250° F. for fifteen minutes to two hours, washing the scoured textiles with water, partially bleaching the washed textiles in an aqueous solution of sodium hypochlorite having a pH of between 9 and 12, at a temperature of between 50° F. to 150° F. for between one-half hour to two hours, souring the partially bleached textiles with an aqueous solution of sodium bisulfite having a pH of between 1 to 5, at a temperature between 50° F. to 120° F. for between five to thirty minutes, washing the textiles from the bisulfite sour in water, and bleaching the washed textiles in an aqueous solution of hydrogen peroxide having a pH of between 8.5 to 11, at temperatures of between 70° F. to 250° F. for between one to three hours.

2. The method of claim 1, wherein the sodium carbonate concentration of the aqueous scouring solution is about 0.5 percent by weight and the ammonium hydroxide concentration of said solution is between 0.8 and 1.1 percent by weight.

3. The method of claim 2, wherein the aqueous hydrogen peroxide solution contains between 0.2 and 1.4 percent H_2O_2 by weight of solution.

4. The method of claim 1, wherein said textiles are scoured with an aqueous solution of sodium hydroxide and ammonium hydroxide.

5. The method of claim 4, wherein the sodium hydroxide concentration of the aqueous scouring solution is about 0.5 percent by weight and the ammonium hydroxide concentration of said solution is between 0.8 and 1.1 percent by weight.

6. The method of claim 3, wherein the aqueous sodium hypochlorite solution contains below 2.5 percent by weight sodium hypochlorite.

7. A method of bleaching textiles containing fibers selected from the group consisting of cotton and linen comprising contacting said fibers with an aqueous solution of from 0.1 to about 2.0 percent by weight of the solution of a member of the group consisting of sodium carbonate and sodium hydroxide, said solution containing ammonium hydroxide in a concentration of from 0.1 to 1.3 percent by weight of the solution and being at a pH of between 9 and 11.5 and at temperatures of between 160° F. to 250° F., partially bleaching the fibers after said contact with an alkaline solution of sodium hypochlorite having a pH of between 9 and 12, souring the partially bleached fibers with an aqueous bisulfite solution having a pH between 1 to 5, and contacting the bisulfite treated fibers with an aqueous solution of hydrogen peroxide having a pH between 8.5 and 11.

8. A method of treating textiles containing fibers of the group consisting of cotton and linen and having dyed portions which bleed in peroxide bleaching solutions comprising contacting said textiles with an aqueous solution of from 0.1 to about 2.0 percent by weight of the solution of a member selected from the group consisting of sodium carbonate and ammonium hydroxide in a concentration of from 0.1 to 1.3 percent by weight of the solution at a pH of between 9 and 11.5 and at temperatures between 160° F. to 250° F., partially bleaching the textiles after said contact with an alkaline solution of sodium hypochlorite at a pH of between 9 and 12, souring the partially bleached textiles with an aqueous bisulfite solution having a pH between 1 to 5, and contacting the bisulfite treated goods with an aqueous solution of hydrogen peroxide at a pH of between 8.5 and 11.

9. The method of claim 8 wherein said textiles are scoured with an aqueous solution of sodium hydroxide and ammonium hydroxide.

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